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# EVALUATION OF AN ELECTROCHEMICAL N<sub>2</sub>/H<sub>2</sub> GAS SEPARATOR

## FINAL REPORT

by

R.D. Marshall, R.A. Wynveen  
and J.N. Carlson

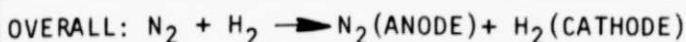
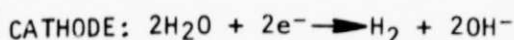
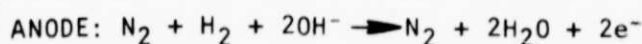
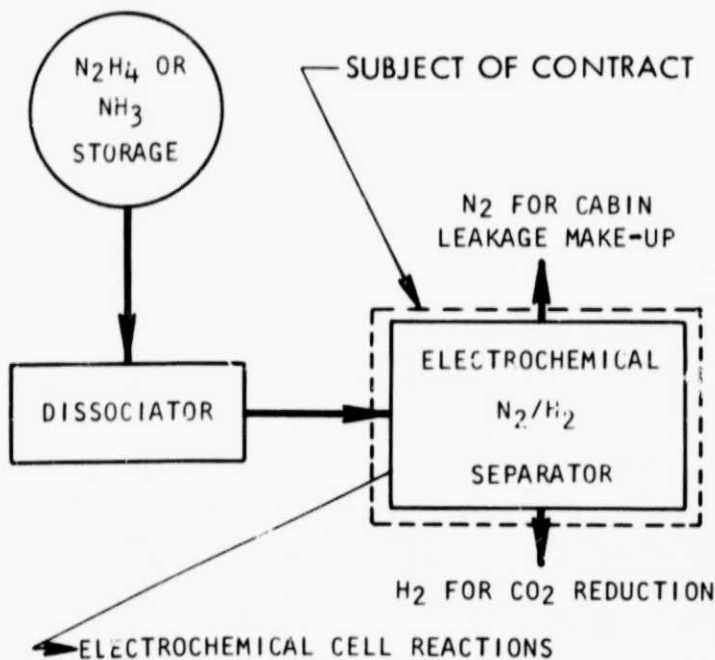
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FOREWORD

The activities described herein, conducted by Life Systems, Inc., were performed under NASA Contract NAS2-7057. J. N. Carlson conducted the evaluation of the palladium-silver approach to  $N_2/H_2$  separation. J. D. Powell designed the test facility electronic controls and instrumentation. J. W. Shumar was responsible for the program's product assurance. The program's technicians were J. C. Bronikowski, G. A. Little, N. S. Sasso, J. J. Palagyi, M. L. Kruszynski and R. A. Clark. Clerical work was completed by C. A. Lucas. The overall Program Manager was R. D. Marshall.

The Contract Technical Monitor was P. D. Quattrone, Chief, Environmental Control Research Branch, NASA Ames Research Center, Moffett Field, California.

The evaluation of alternate  $N_2/H_2$  separation techniques was conducted with the assistance of Dr. R. J. Adlef and K. Kacholia at Case Western Reserve University, Cleveland, Ohio.

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## SUMMARY

A program was successfully completed to evaluate an electrochemical nitrogen/hydrogen ( $N_2/H_2$ ) separator for use in a spacecraft nitrogen ( $N_2$ ) generator. For comparison purposes, a literature survey to evaluate alternate separation techniques was conducted. Based on the technical data obtained a  $N_2/H_2$  separator subsystem consisting of an organic polymer gas permeator first stage and an electrochemical second and third stage was estimated to have the lowest total spared equivalent weight, 257 kg (566 lb), for a  $7.88 \times 10^{-5}$  kg/s (15 lb/day)  $N_2$  generation rate.

An analysis of the end-item application defined the  $N_2$  generator subsystem and established the design criteria and subsystem specifications used to evaluate the  $N_2/H_2$  separation techniques. A pre-design analysis of the electrochemical  $N_2/H_2$  separator revealed that its use as a first stage resulted in too high a power requirement to be competitive with the organic polymer membrane and the palladium-silver membrane separation methods. As a result, program emphasis was placed on evaluating the electrochemical second- and third-stage designs for subsequent integration with a nonelectrochemical first stage.

A parametric test program characterized cell performance and established second- and third-stage electrochemical  $N_2/H_2$  separator operating conditions. A design verification test was completed on the second and third stages. The second stage was then successfully endurance tested for 200 hours.

Testing was conducted using a test facility having the necessary electronic controls and instrumentation to regulate and monitor separator performance.

The completed literature survey identified eight alternate  $N_2/H_2$  separation techniques. Of the alternate techniques evaluated,  $N_2/H_2$  separation based on heated palladium-silver diffusion tubes had an estimated total spared system equivalent weight of 317 kg (698 lb) for comparison to the lower equivalent weight polymer-electrochemical separator.

## INTRODUCTION

In spacecraft operation it is necessary to replace cabin air lost through cabin leakage and compartment depressurizations. Although the lost gas is principally a mixture of oxygen ( $O_2$ ) and nitrogen ( $N_2$ ), the make-up gas need not be stored as  $O_2$  and  $N_2$ . The  $N_2$  component of air can be stored as non-cryogenic liquid hydrazine ( $N_2H_4$ ) or liquid ammonia ( $NH_3$ ) and catalytically dissociated to yield a mixture of  $N_2$  and hydrogen ( $H_2$ ). The  $N_2/H_2$  mixture can then be separated by a variety of methods to yield the make-up  $N_2$  and a supply of  $H_2$  for use in other regenerative life support subsystems.

The objective of this program was to evaluate an electrochemical  $N_2/H_2$  separator for comparison with other  $N_2/H_2$  separation methods. The program was divided into four tasks whose specific objectives were to:

1. Conduct an analysis of the end-item application to establish design criteria, detailed subsystem specifications and operating environment, and to perform the initial pre-cell design analysis with the results to serve as the basis for subsequent designs.
2. Design, fabricate, assemble and test a single cell electrochemical  $N_2/H_2$  separator to establish the baseline operating conditions for the second and third stages of an electrochemical  $N_2/H_2$  separator.
3. Design, fabricate, assemble and test two modules representing the second and third stages of a  $N_2/H_2$  separator.
4. Conduct a comparison study of alternate methods of  $N_2/H_2$  separation to determine the optimum method for separating pure  $N_2$  from a  $N_2/H_2$  mixture.

The objectives of the program tasks were met and the design recommendations for a  $N_2/H_2$  separator for use in a nominal  $7.88 \times 10^{-5}$  kg/s (15 lb/day)  $N_2$  generator were prepared. The following sections summarize the results of the program.

#### END ITEM APPLICATION ANALYSIS

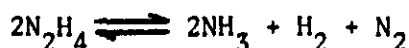
An end item application analysis was completed to define

1. the  $N_2$  Generator Subsystem,
2. the  $N_2/H_2$  separator design requirements for use in the  $N_2$  Generator Subsystem, and
3. the design evaluation criteria for evaluating  $N_2/H_2$  separation techniques.

#### $N_2$ Generator Subsystem

For long-term space missions a nominal  $7.88 \times 10^{-5}$  kg/s (15 lb/day)  $N_2$  generator will be required to make up for losses resulting from cabin leakage and depressurizations.

The  $N_2$  Generation Subsystem shown in Figure 1 is based on  $N_2H_4$  as the storable form of  $N_2$ . Liquid  $N_2H_4$  at room temperature is fed into the dissociator where it is catalytically cracked in two consecutive reactions (1):



(1) All references are cited at the end of the report.

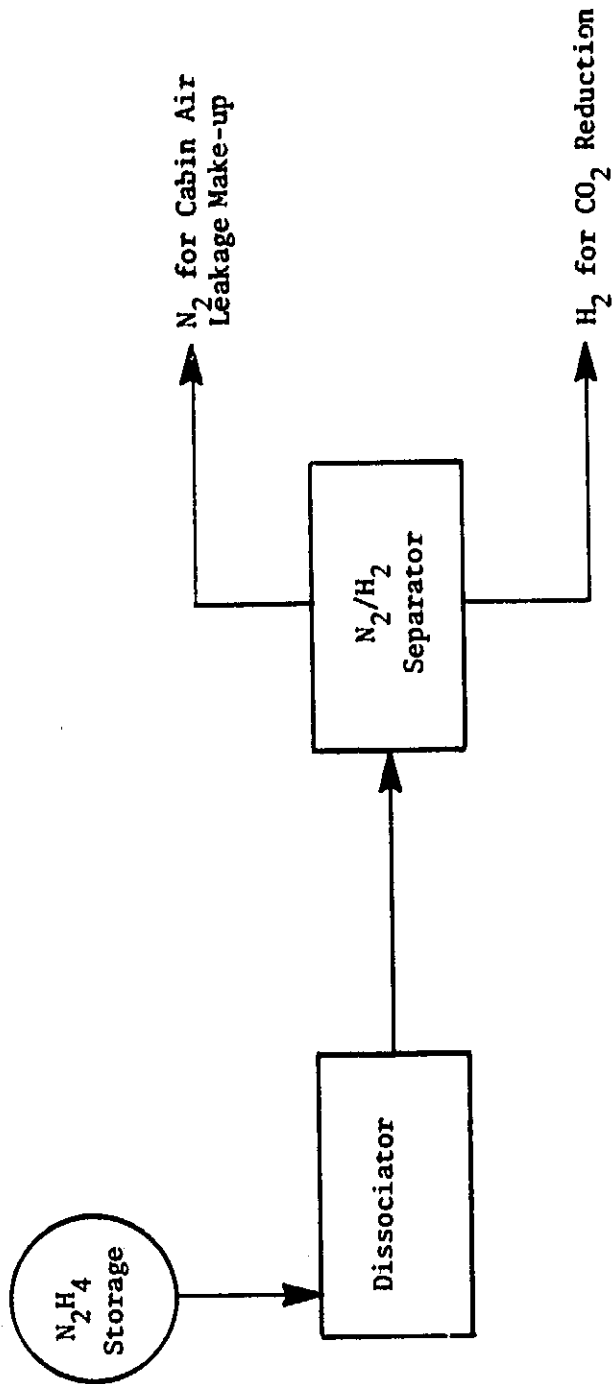


FIGURE 1 BLOCK DIAGRAM OF A  $N_2$  GENERATION SUBSYSTEM

The  $N_2/H_2$  mixture containing traces of unreacted  $N_2H_4$  and  $NH_3$  leaves the dissociator at approximately 800K (980F) and is cooled in a heat exchanger before entering the  $N_2/H_2$  separator. The amount of cooling required depends on the operating temperature of the  $N_2/H_2$  separator. The separated  $N_2$  and  $H_2$  product streams are then brought to room temperature for storage or use. If the separator operates at room temperature to 322K (120F) no cooling of the product streams is required.

Ammonia could be substituted for  $N_2H_4$  yielding more  $H_2$  per weight of  $N_2$  but the  $NH_3$  dissociation process is more complicated and requires temperatures above 1033K (1400F).

### Design Requirements

The design requirements for integrating the  $N_2/H_2$  separator into a  $N_2$  Generator Subsystem are detailed in Table 1.

### Design Evaluation Criteria

Various methods are available for separating  $N_2/H_2$  mixtures. The optimum approach for future NASA Manned Flight Programs requires the selection of a cost-effective method, i.e., one which considers development time and available funds.

Table 2 lists the quantitative, semi-quantitative and qualitative design evaluation criteria subsequently used to evaluate the various methods of  $N_2/H_2$  separation considered during the program.

### ELECTROCHEMICAL SEPARATION OF $N_2/H_2$

An analytical study of the electrochemical separation of  $N_2/H_2$  mixtures was completed to

1. define the electrochemical  $H_2$  removal process,
2. prepare a preliminary design and total equivalent weight estimate, and
3. analyze methods for meeting power, heat removal, and humidification requirements.

### Process Description

Pure  $H_2$  is separated from a  $N_2/H_2$  mixture in a cell consisting of two porous metal electrodes separated by an electrolyte solution of aqueous potassium hydroxide (KOH). The electrolyte is held in an absorbent porous material which prevents the solution from leaking through the electrodes. Compartments adjacent to the electrodes provide passageways for distributing the  $N_2/H_2$  mixture and for collecting the separated  $H_2$ .

TABLE 1 DESIGN REQUIREMENTS

**N<sub>2</sub>/H<sub>2</sub> Feed**

Source	Cracked N <sub>2</sub> H <sub>4</sub>
N <sub>2</sub> Flow	$7.88 \times 10^{-5}$ kg/s (15 lb/day)
H <sub>2</sub> Flow	$1.12 \times 10^{-5}$ kg/s (2.14 lb/day)
Temperature	294-800K (70-980F)
Pressure	$6.21-10.3 \times 10^5$ N/m <sup>2</sup> (90-150 psia)
Impurities	<1% NH <sub>3</sub> and <1% N <sub>2</sub> H <sub>4</sub>

**N<sub>2</sub> Product**

Delivery Rate	$7.88 \times 10^{-5}$ kg/s (15 lb/day)
Composition	<0.1% H <sub>2</sub>
Temperature	294K (70F)
Pressure	$>5.86 \times 10^5$ N/m <sup>2</sup> (85 psia)

**H<sub>2</sub> Product**

Delivery Rate	$1.12 \times 10^{-5}$ kg/s (2.14 lb/day)
Composition	<5% N <sub>2</sub> impurities
Temperature	294K (70F)
Pressure	$>1.72 \times 10^5$ N/m <sup>2</sup> (25 psia)

TABLE 2 DESIGN EVALUATION CRITERIA<sup>(a)</sup>

Quantitative Criteria

1. Basic system launch weight.
2. Basic system launch volume.
3. System power requirements.
4. Expendable weight (logistic costs).
5. Expendable volume.
6. Spares weight.
7. Spares volume.
8. Heat rejection.
9. Resupply weight (expendables and spares).

Semiquantitative Criteria

1. Development cost (nonrecurring).
2. Interface sensitivity.
3. Amount of instrumentation (control/monitoring).
4. Crew time for maintenance.
5. Number of major subsystem components.

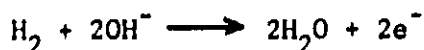
Qualitative Criteria

1. Relative development risk (available for flight by 1978).
2. Noise level.
3. Growth potential.
4. Conformance to 5-year operating life.
5. Flexibility to change in mission requirements.
6. Tolerance to maximum (12-month) quiescent operation.

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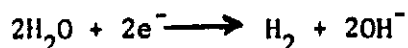
(a) These criteria assume that all methods considered have already passed the test based on ability to perform the function in a manner not hazardous to crew or equipment.

The  $N_2/H_2$  mixture is fed into the anode compartment of the cell. When a DC power supply is connected to the cell electrodes, the  $H_2$  within the gaseous mixture reacts with hydroxyl ions ( $OH^-$ ) within the electrolyte to form water ( $H_2O$ ) and electrons. The equation for the anode half cell reaction is:



The  $H_2O$  released at the anode remains in the electrolyte and the electrons travel from the anode to the cathode through the external circuit containing the power supply.

At the cathode the electrons react with  $H_2O$  forming  $H_2$  and  $OH^-$ . The equation for the cathode half cell reaction is:



The  $OH^-$  migrate from the cathode to the anode under the electromotive force provided by the power supply for reaction with more  $H_2$ .

The  $H_2$ -depleted  $N_2/H_2$  stream at the anode and the  $H_2$  generated at the cathode exit through the exhaust manifolds of the cell. The process continues as long as  $H_2$  and power are provided, and cell moisture balance is maintained within limits.

#### Current Required

According to Faraday's Law of Electrolysis, the quantity of  $H_2$  consumed from the gaseous mixture and released in the cathode compartment is directly proportional to the current (electrons) allowed to flow through the cell. The output of a unit can be expressed by the equation:

$$W = 1.04 \times 10^{-8} (I) (N)$$

where:

$W$  =  $H_2$  output, kg/s

$I$  = current, A

$N$  = number of series connected cells in the unit

The current required to remove all the 0.97 kg (2.14 lb) of  $H_2$  obtained from the decomposition of  $N_2H_4$  to yield  $7.88 \times 10^{-5}$  kg  $N_2$ /s (15 lb  $N_2$ /day) corresponds to 1077 A.

#### Power Required

When the electrochemical  $N_2/H_2$  separator is put on load, each electrode polarizes thereby increasing cell voltage. The power required by the separator



can be expressed by the equation:

$$P = (E)(I)(N)$$

where:

P = power required, W

E = cell voltage, V

I = current, A

N = number of series connected cells in the stack

The power required to remove 0.97 kg (2.14 lb) of  $H_2$  at a cell voltage of 0.40 V is 430.8 W.

#### Preliminary Design Analysis

Table 3 presents an equivalent weight estimate of a three-stage electrochemical  $N_2/H_2$  separator based on  $N_2H_4$  as the storable form of  $N_2$ . The high equivalent weight penalty associated with the first stage is indicated. As a result, the first stage separator was replaced with a nonelectrochemical process identified during the studies of alternate separation techniques (see page 30). The second- and third-stage electrochemical  $N_2/H_2$  separators, however, were retained because of their attractive equivalent weights.

#### Power Requirements

A study was made to determine if the high power equivalent weight penalty associated with the first-stage electrochemical  $N_2/H_2$  separator could be minimized using unregulated spacecraft power or power cycling.

Regulated Versus Unregulated Power. None of the stages of an electrochemical  $N_2/H_2$  separator integrate with the available unregulated power supplies. The low voltage and high current density of each stage would require an excessive number of cells (>100) to integrate with the low weight penalty unregulated 112 VDC, 56 VDC or 28 VDC power.

Continuous Versus Sunlit Side Only Operation. The electrochemical  $N_2/H_2$  separator is compatible with power cycling, sunlit side operation only. The second and third stages of the electrochemical  $N_2/H_2$  separator, however, can be operated continuously because of their small relative power requirements (Table 3).

#### Heat Removal

All the power required to drive the electrochemical  $N_2/H_2$  separator is dissipated as heat. Any heat removal technique applicable to spacecraft can be

TABLE 3 EQUIVALENT WEIGHT ESTIMATE OF A THREE-STAGE  $N_2/H_2$  SEPARATOR  
WITH  $N_2H_4$  AS THE  $N_2/H_2$  SOURCE

Stage	% H <sub>2</sub> Removed	No. Cells	Current per A	Power, W	Heat Load, W	WEIGHT PENALTIES (2)			
						Module Weight, kg (lb)	Power Penalty, kg (lb)	Heat Load, kg (lb)	Total Equivalent Weight, kg (lb)
1 (c)	90	20	48.5	359	359	9.1 (20)	96.2 (212)	71.1 (157)	176.4 (389)
2 (d)	9	3	32.2	16	16	4.1 ( 9)	4.3 (9.5)	3.2 ( 7)	11.6 ( 26)
3 (e)	0.9	1	9.7	1	1	2.3 ( 5)	0.3 (0.2)	0.2 (0.4)	2.8 ( 6)
									190.8 (420)
Accessories									22.7 ( 50)
Total (unspared)									213.5 (470)

(a) Continuous regulated DC power at 0.268 kg/W (591 lb/kw).

(b) Heat rejected to cabin air at 0.198 kg/W (0.128 lb/Btu/hr).

(c) 538 mA/cm<sup>2</sup> (500 ASF) at 0.37 V and 90.7 cm<sup>2</sup> (0.10 ft<sup>2</sup>) electrodes.

(d) 144 mA/cm<sup>2</sup> (134 ASF) at 0.17 V and 223 cm<sup>2</sup> (0.24 ft<sup>2</sup>) electrodes.

(e) 44 mA/cm<sup>2</sup> (40 ASF) at 0.10 V and 223 cm<sup>2</sup> (0.24 ft<sup>2</sup>) electrodes.

used. The second- and third-stage separators have only a small heat removal requirement (Table 3).

#### Humidification Requirements

The concentration of the electrolyte inside an electrochemical cell is a direct function of the operating temperature. To maintain the concentration at a fixed level, the moisture content of the gas streams must be balanced such that there is no net transfer of  $H_2O$  into or out of the separator.

The  $N_2/H_2$  feed gas initially contains no moisture. The product gases leaving the separators, however, do. Make-up  $H_2O$ , therefore, must be added to the feed gases to maintain the cell's electrolyte concentration.

The following techniques were identified to minimize gas humidification requirements:

1. Start with hydrated or partially hydrated  $N_2H_4$  ( $N_2H_4 \cdot H_2O$ ) instead of anhydrous  $N_2H_4$ .
2. Pass the  $N_2/H_2$  mixture through a  $H_2O$  humidifier prior to entering the electrochemical separator.
3. Operate at elevated pressures.
4. Operate at room temperature.
5. Operate at higher electrolyte concentrations.
6. Mix some hydroxylamine ( $NH_2OH$ ) with the  $N_2H_4$  prior to dissociation.
7. Use a lower  $H_2$ -bearing compound instead of  $N_2H_4$ .

Techniques 1, 2 and 6 could be used to eliminate the need for humidification.

#### HARDWARE DESCRIPTION

The hardware designed, fabricated and used in the experimental characterization of an electrochemical  $N_2/H_2$  separator consisted of

1. the test facility capable of testing single cells and multicell modules, and having the capability of breadboarding a second- and third-stage electrochemical separator,
2. the single cell used for the parametric characterization test program, and
3. the scaled-up module cell used in the second and third stage design verification and endurance test programs.

### Test Facility

The test stand used in the  $N_2/H_2$  separator testing is shown in Figure 2. Pure  $N_2$  and  $H_2$  can be continuously mixed in the desired ratio by regulating the feed pressure and flow of the pure gases as shown in the system flow schematic, Figure 3. The  $N_2/H_2$  mixture is then humidified in a saturator tank before entering the electrochemical separator. The exiting gas streams from the separator are dehumidified in a condenser and vented.

Pressure Control. The  $H_2$  delivery pressure is controlled by the backpressure regulator on the  $H_2$  vent stream. The  $N_2$  delivery pressure is controlled with respect to the  $H_2$  delivery pressure using a differential pressure regulator, a  $N_2$  signal stream and a backpressure regulator on the  $N_2$  vent stream. The differential pressure regulator is referenced to the  $H_2$  delivery pressure which in turn controls the signal pressure to the  $N_2$  backpressure regulator. As the  $H_2$  delivery pressure changes, the signal pressure to the  $N_2$  backpressure regulator changes to maintain a set differential pressure. The differential pressure control allows the  $N_2$  pressure to be maintained between  $-1.03 \times 10^5$  N/m<sup>2</sup> and  $+0.90 \times 10^5$  N/m<sup>2</sup> (15 psid and +13 psid) with respect to the  $H_2$  pressure.

Temperature Control. The cell, the humidifier temperature and the  $N_2/H_2$  feed mixture temperature are individually controlled to maintain cell operating temperature and moisture balance. The operating temperature of the separator is controlled by blowing ambient air over the external fin portion of the cell current collectors. The humidifier temperature is controlled by a heater and a coolant coil which are part of the saturator tank. The  $N_2/H_2$  feed mixture temperature is controlled by heating tape which is wrapped around the  $N_2/H_2$  feed lines.

Power Supply. The stand is equipped with a power supply that allows the electrochemical separator to be run at either constant current (0-66 A, adjustable) or constant voltage (0-20 V, adjustable).

Instrumentation. The stand provides for measuring individual cell voltages, current,  $N_2$  and  $H_2$  flows, temperatures and pressures. In addition, the system is protected by four adjustable shutdown modes. They are

1. excessive differential pressure,
2. excessive operating temperature,
3. excessive operating pressure, and
4. excessive individual cell voltage (from 1 to 4 cells).

When a shutdown mode occurs, the  $N_2$  and  $H_2$  feed gas streams and power supply are shut off automatically.

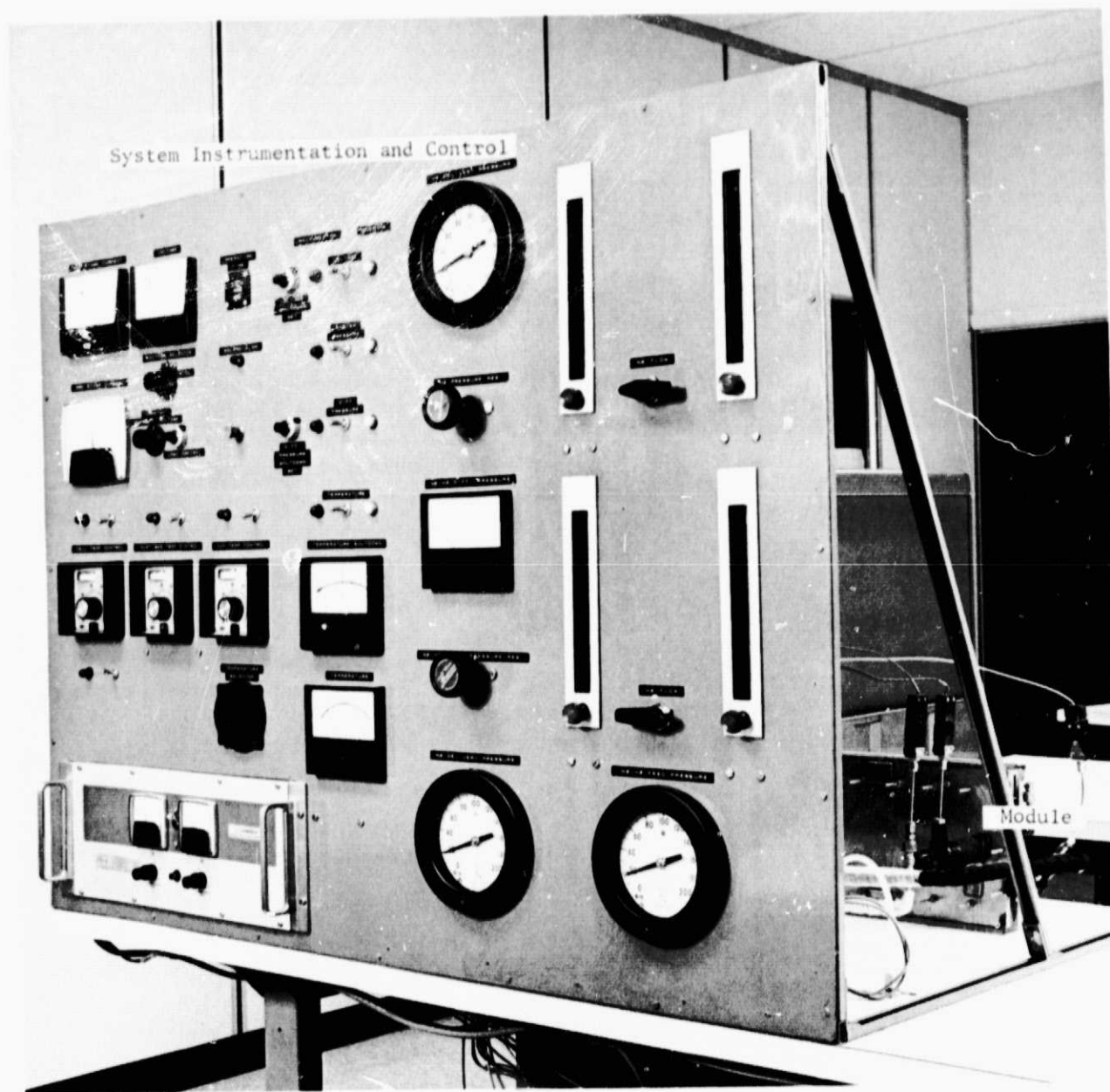


FIGURE 2 ELECTROCHEMICAL  $N_2/H_2$  SEPARATOR TEST FACILITY

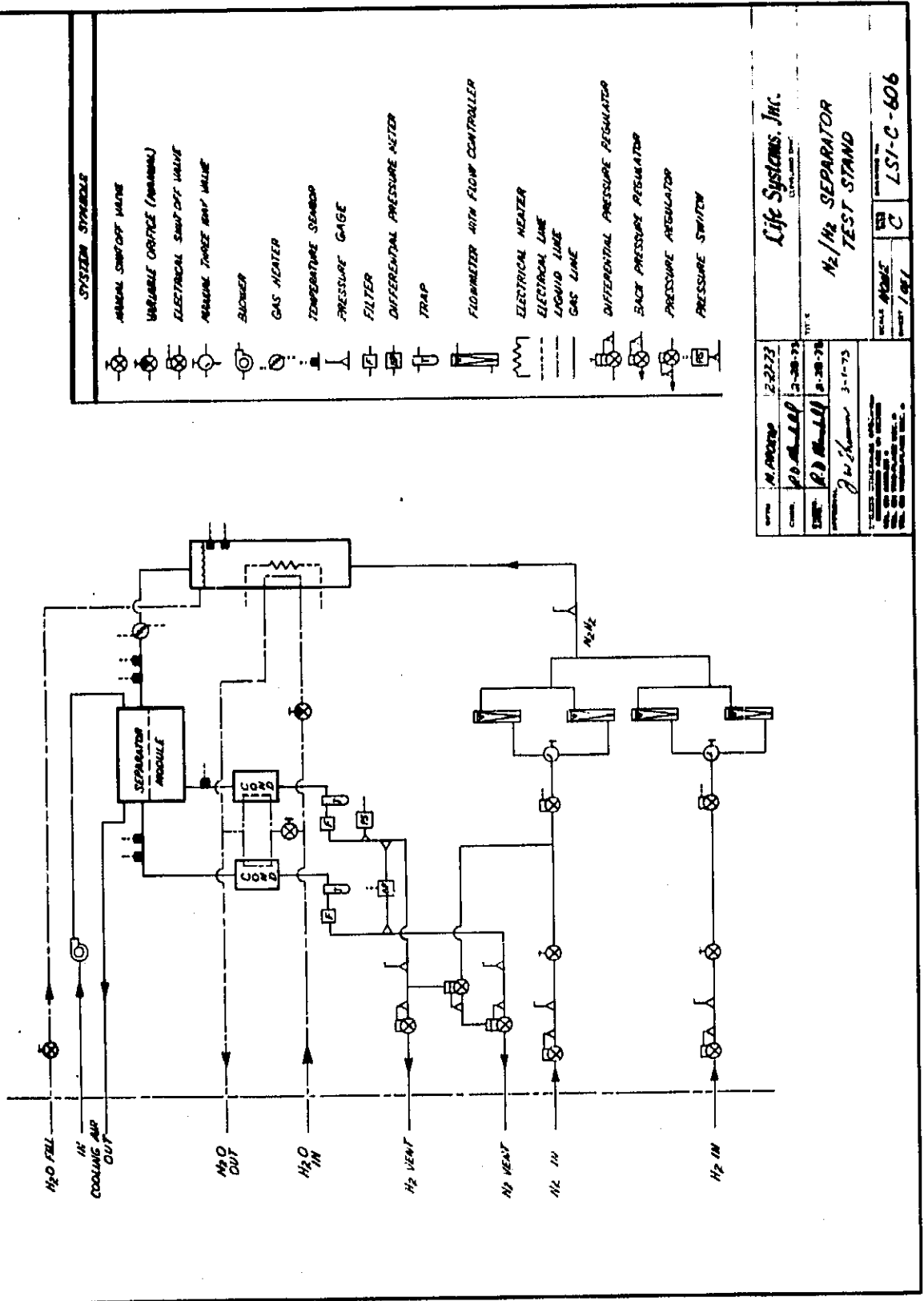


FIGURE 3 TEST STAND FLOW SCHEMATIC

Laboratory Breadboard Features. The test facility is capable of breadboarding the second and third stages of an electrochemical  $N_2/H_2$  separator.

The breadboard controls allow the second stage to operate at constant current while the third stage is controlled at constant voltage. Pressure and temperature controls are the same as in single-stage operation.

## Single Cell Design

Figures 4 and 5 are the schematic and photograph of the single cell used in the Parametric Test Program.

The  $N_2/H_2$  feed is manifolded through the anode frame into the anode cavity where it is dispersed over the electrode surface by the gas cavity spacer. The  $H_2$  is then electrochemically removed and the  $H_2$ -depleted  $N_2$  stream is exhausted from the cell through the anode frame. At the cathode,  $H_2$  is uniformly generated at the electrode surface and exhausts the cell through the cathode frame.

The single-cell characteristics and materials of construction are given in Table 4.

## Module Cell Design

Figures 6 and 7 are the schematic and photograph of the module cell used in testing the second- and third-stage electrochemical  $N_2/H_2$  separator. The design allows for manifolding of the gases similar to that in the single-cell design. Since the second stage consists of three cells, the module cell design allows several cells to be stacked together. Not shown in the schematic is the insulation plate and endplate at each end of the cell stack. The electrical connections of the second-stage module are somewhat different from that of a single cell. The anode of the first cell and the cathode of the last cell are connected to the power supply and the current passes directly through the cell stack without external electrical connections.

The module-cell characteristics and materials of construction are given in Table 4.

## TEST PROGRAM

The test program was divided into four areas:

1. Calibration Tests
2. Single-Cell Parametric Tests
  - Effect of Temperature
  - Effect of  $N_2:H_2$  Feed Ratios
  - Effect of Pressure

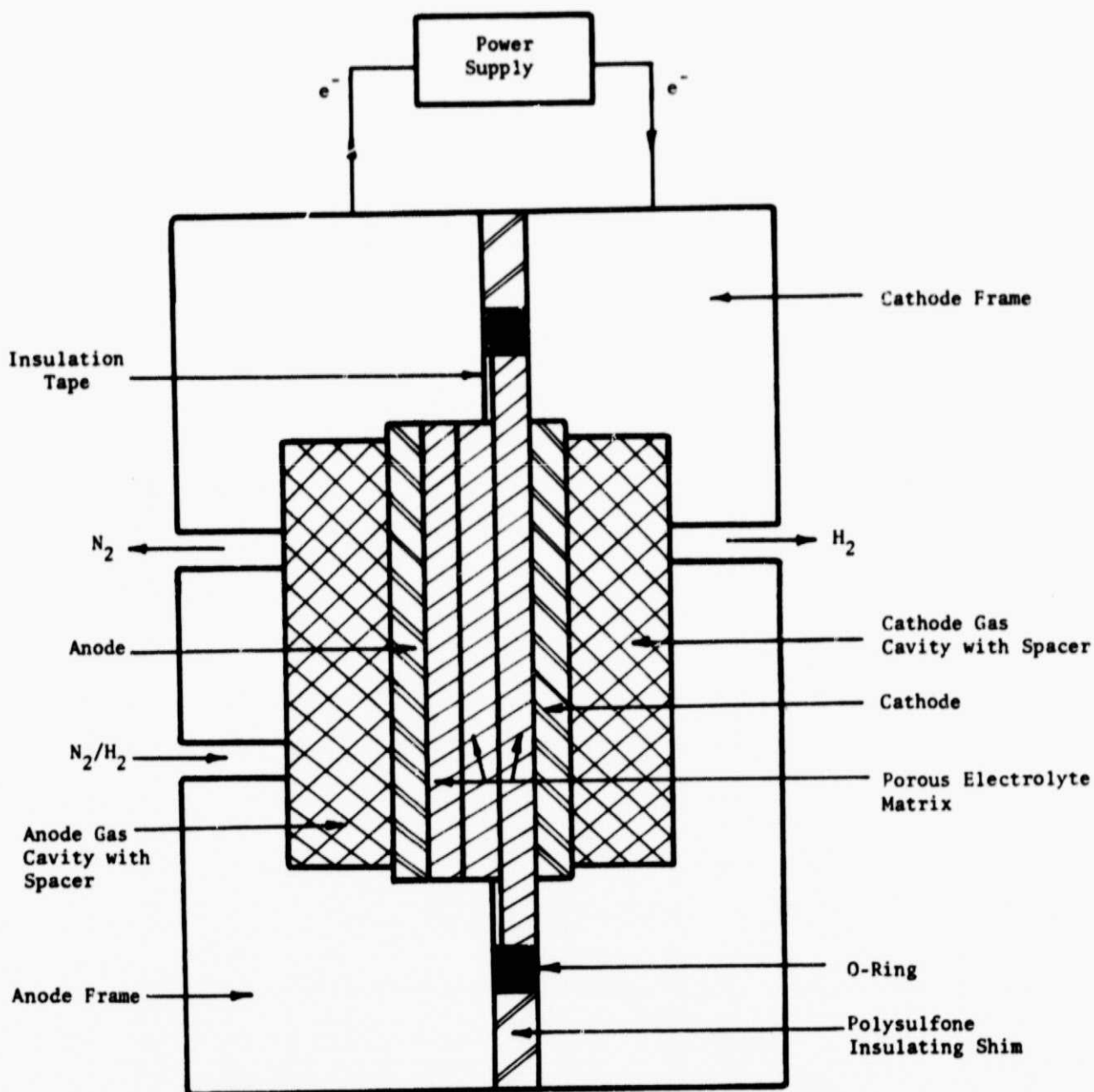


FIGURE 4 SINGLE-CELL SCHEMATIC



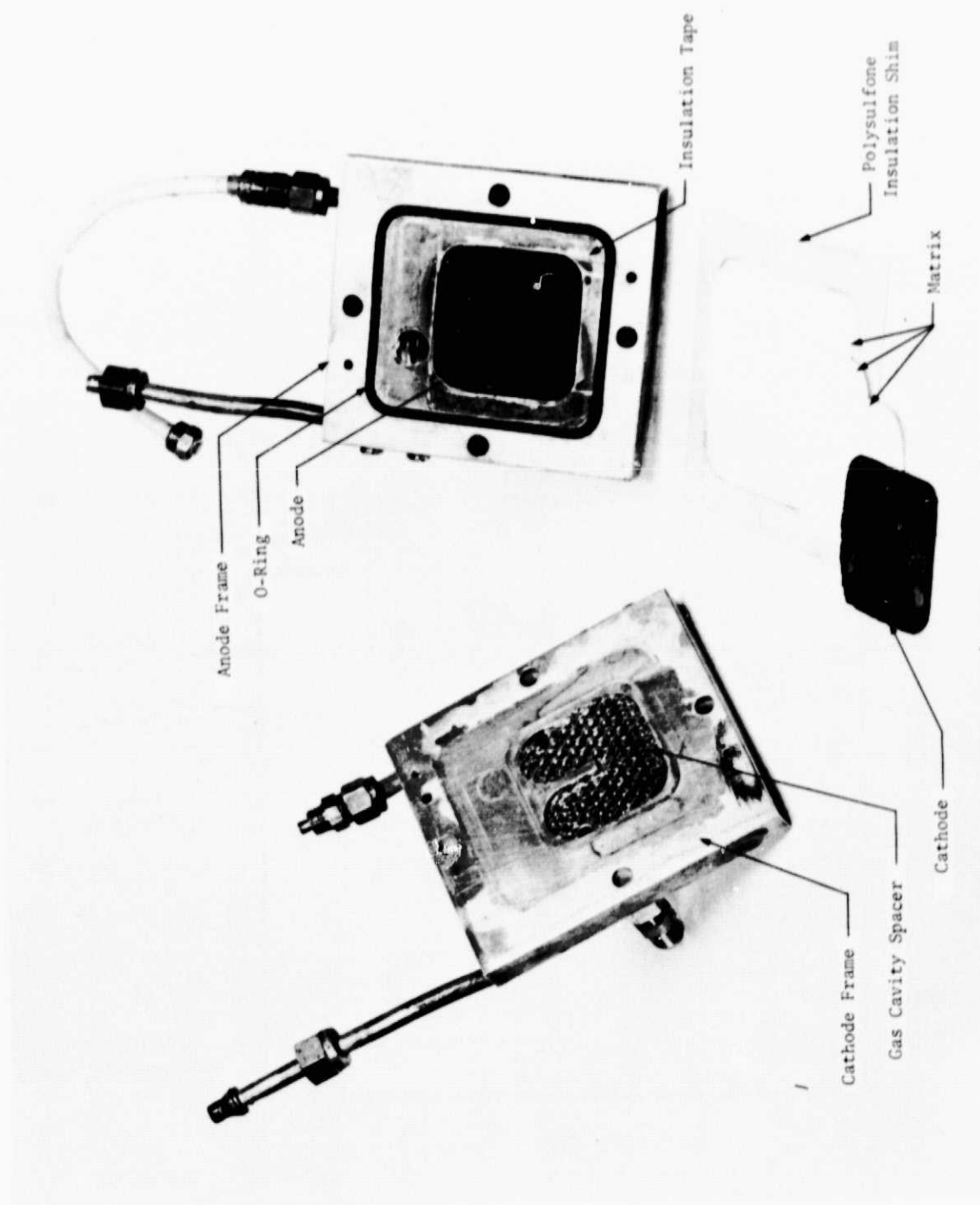


FIGURE 5 EXPERIMENTAL SINGLE-CELL

TABLE 4 SINGLE-CELL AND MODULE-CELL CHARACTERISTICS

	Single Cell	Module Cell
Anode Frame	Stainless Steel	Polysulfone
Cathode Frame	Stainless Steel	Polysulfone
Gas Cavity Spacer	Nickel Exmet	Gold-Plated Silver Exmet
Current Collectors	Not Required	Gold-Plated Silver
O-Rings	Ethylene Propylene	Ethylene Propylene
Electrode Gaskets	Not Required	Ethylene Propylene
End Plates	Stainless Steel	Stainless Steel
Insulation Plates	Polysulfone	Polypropylene
Insulation Tape	Teflon	Not Required
Matrix Material	LSI Proprietary	LSI Proprietary
Matrix Thickness	0.076 cm (0.030 in)	0.076 cm (0.030 in)
Electrode	LSI Proprietary	LSI Proprietary
Electrode Area	22.3 cm <sup>2</sup> (0.024 ft <sup>2</sup> )	223 cm <sup>2</sup> (0.24 ft <sup>2</sup> )
Electrode Thickness	0.025 cm (0.010 in)	0.025 cm (0.010 in)
Electrolyte	25% KOH	25% KOH

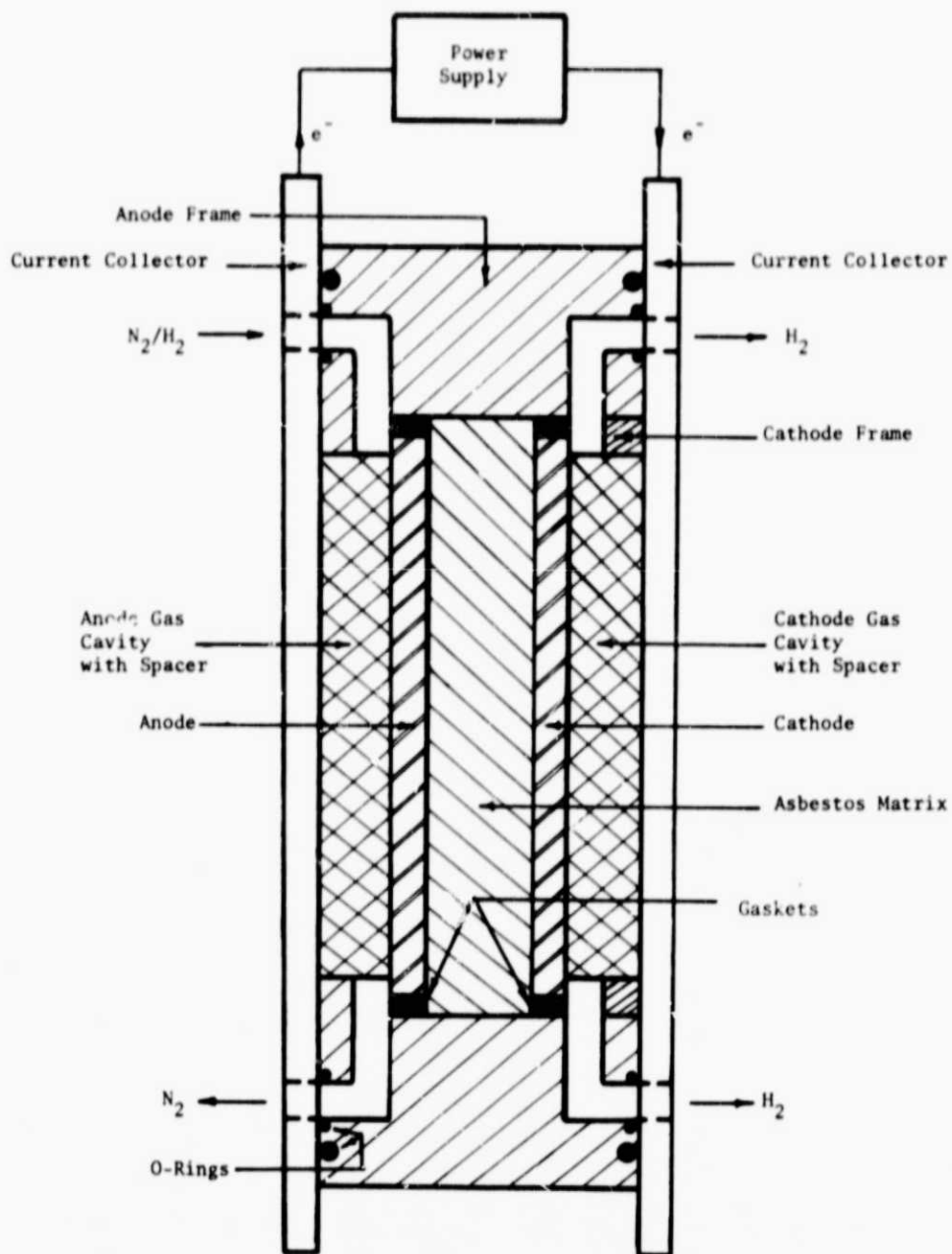


FIGURE 6 MODULE-CELL SCHEMATIC

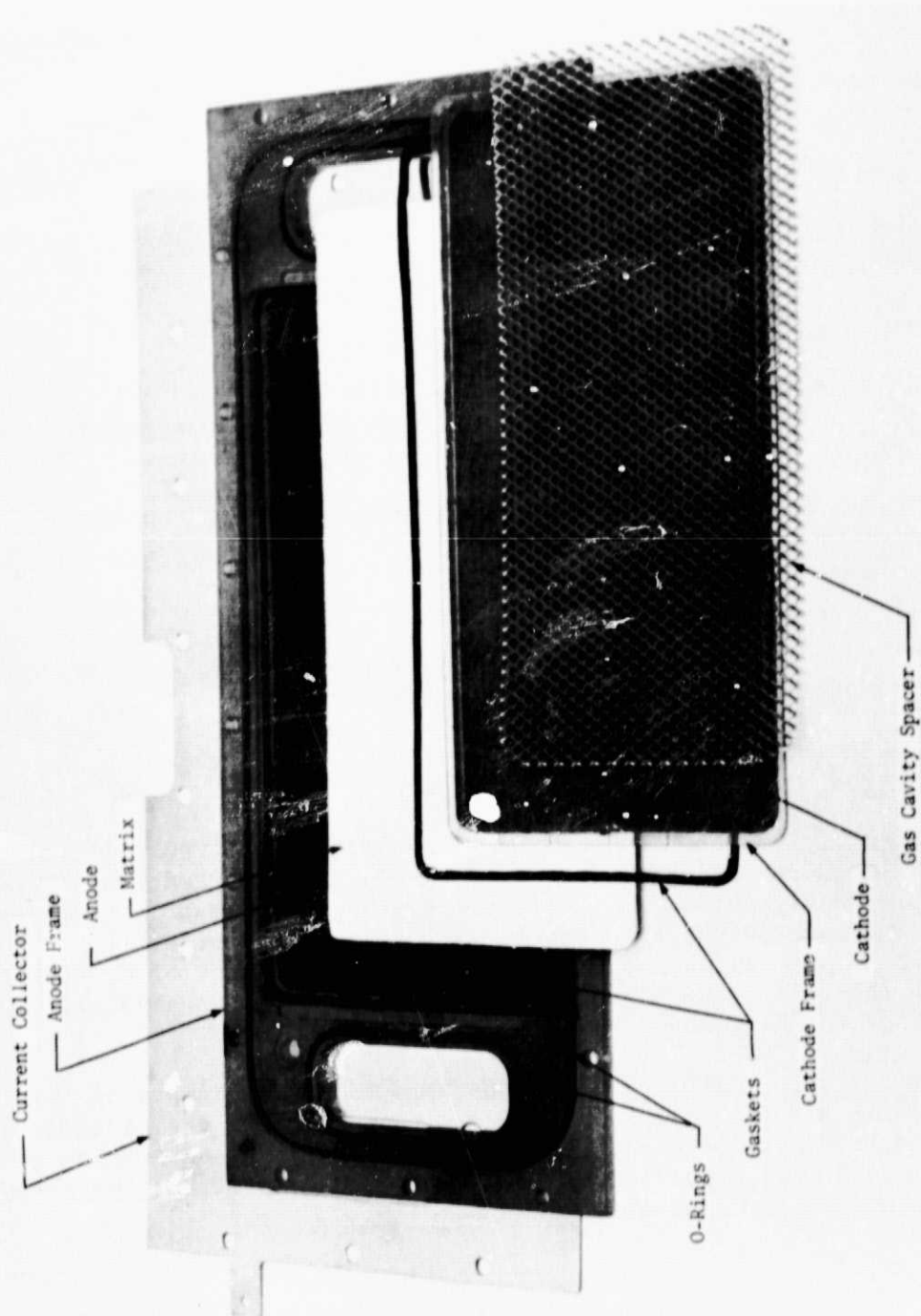


FIGURE 7 FULL-SIZED CELL FOR STAGES 1, 2 AND 3

Effect of Differential Pressure  
Effect of Trace Contaminants

3. Second-Stage Tests

Shakedown Test  
Design Verification Test  
Endurance Test

4. Third-Stage Tests

Shakedown Test  
Design Verification Test

Calibration Tests

Product Assurance specified that calibration tests be carried out on all test facility parameter-monitoring equipment; flowmeters, pressure gages, ammeters, voltmeters and pressure transducers. These calibrations were completed.

In addition, the shutdown set-point controls were calibrated based on simulating levels at the sensor.

Single Cell Parametric Test Program

The parametric test program was successfully completed. Table 5 summarizes the range over which the parameters tested were varied.

Performance Measurements. The performance of an electrochemical  $N_2/H_2$  separator is described by the electrical efficiency and the  $H_2$  removal efficiency.

The electrical efficiency of the separator is reflected by the cell voltage. Since the cell requires electrical power to perform the separation, a low cell voltage reflects a high electrical efficiency. The  $H_2$  removal efficiency is reflected by the maximum current density ( $i_{max}$ ) at which the cell operates.<sup>(a)</sup> The  $i_{max}$  at which the cell can operate determines the amount of  $H_2$  that can be removed by the cell. The higher  $i_{max}$  for a given  $H_2$  concentration and feed rate, the better the  $H_2$  removal efficiency.

Effect of Temperature. Figure 8 shows the effect of temperature on cell performance for 327K and 361K (130F and 190F). As expected, the electrical efficiency and  $H_2$  removal efficiency increased with increased temperature. It should be noted that as cell operating temperature increases the humidification requirements of the cell also increases.

(a) As current density increases cell voltage increases. At some current density the cell voltage will rise sharply, indicating that there is insufficient  $H_2$  available to maintain the current.

TABLE 5 SUMMARY OF PARAMETRIC PERFORMANCE CHARACTERIZATION CURVES

Parameter Varied	Range	Cell Voltage	Temp, K (F)	Pressure	$N_2/H_2$ Flow, $\frac{cm^3}{s}$	Current, $\frac{mA}{cm^2}$ (ASF)
Cell Temperature	327 to 361K (130 to 190F)	Figure 6	Var (a)	Amb (b)	2.55	Var.
$N_2:H_2$ Ratio	1:3 to 1:0.1	Figure 7	361 (190)	Amb	Var	Var
System Pressure	Amb to $6.21 \times 10^5 N/m^2$ (Amb to 90 psia)	Figure 8	361 (190)	Var	4.38	Var
$N_2-H_2$ Differential Pressure	$-6.9$ to $20.7 \times 10^3 N/m^2$ (-1 to 3 psid)	Figure 9	361 (190)	Amb	4.62	Var
$N_2H_4$ Contaminant	1%	Figure 10	342 (156)	Amb	6.8	108 (100)
$NH_3$ Contaminant	1%	Figure 11	342 (156)	Amb	6.8	108 (100)

(a) Var = Variable

(b) Amb = Ambient pressure, 96.0 to 98.7  $\times 10^3 N/m^2$  (13.9 to 14.3 psia)

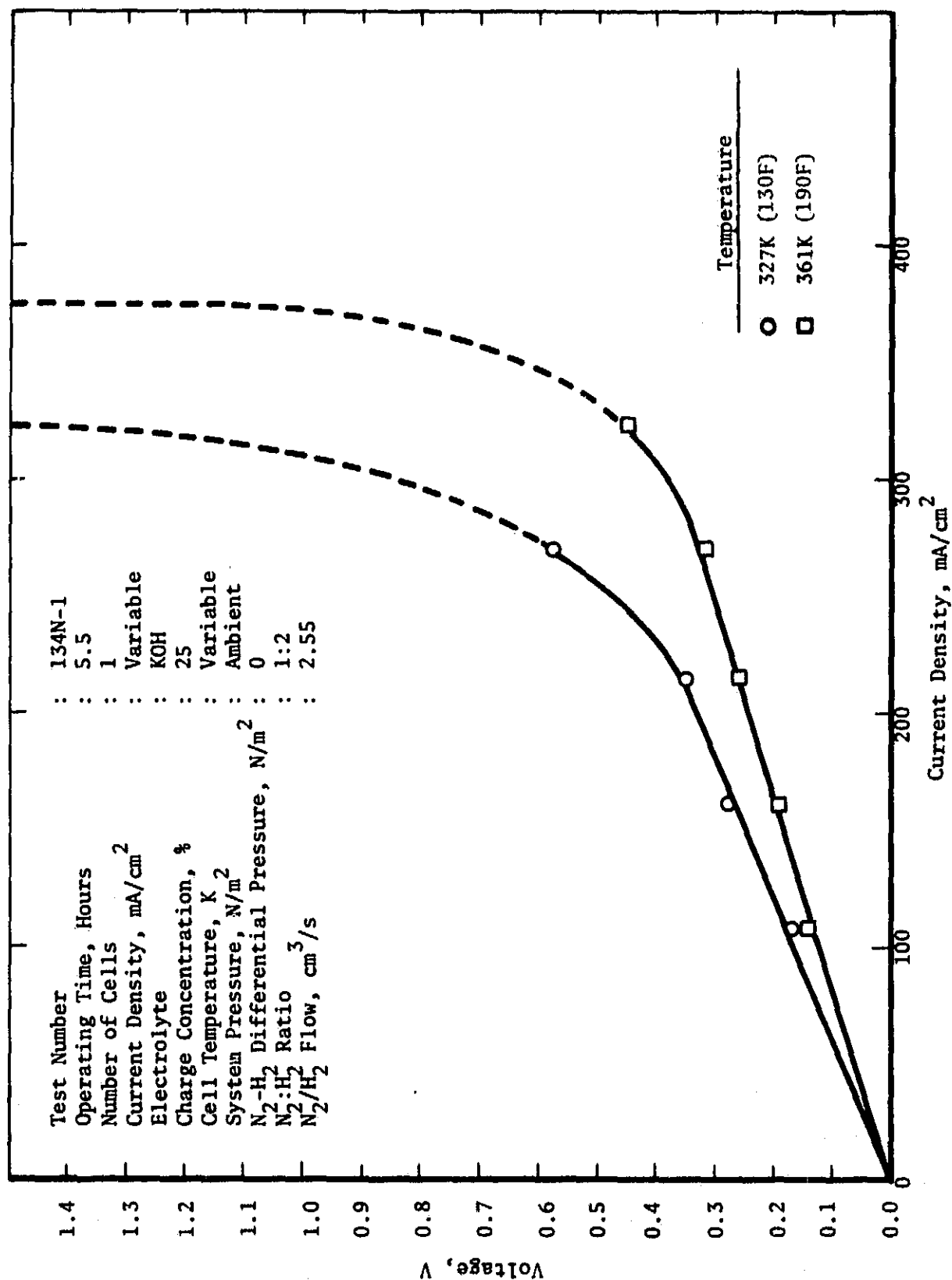


FIGURE 8 EFFECT OF TEMPERATURE ON CELL PERFORMANCE

Effect of  $N_2:H_2$  Feed Ratios. Figure 9 shows the effect of  $N_2:H_2$  feed ratios on cell performance for ratios of 1:3, 1:2.1, 1:1.3, 1:0.5, 1:0.2 and pure  $H_2$ . As the ratio of  $N_2$  to  $H_2$  decreased (i.e., greater amounts of  $H_2$ ) both electrical efficiency and  $H_2$  removal efficiency increased.

Effect of Pressure. Figure 10 shows the effect of operating pressure on cell performance for ambient and  $6.21 \times 10^5 \text{ N/m}^2$  (90 psia). As the cell operating pressure increased, both the electrical and  $H_2$  removal efficiency increased. The higher pressure has a secondary advantage; it reduces the humidification requirements.

Effect of Differential Pressure. Figure 11 shows the effect of differential pressure on cell performance for  $N_2$  to  $H_2$  differential pressures of  $-6.9 \times 10^3 \text{ N/m}^2$  to  $+207 \times 10^3 \text{ N/m}^2$  (-1 to +3 psid). As the  $N_2$  to  $H_2$  differential pressure increased, the electrical and  $H_2$  removal efficiency increased. It should be noted that as the absolute operating pressure of the cell increases, the effect of  $N_2-H_2$  differential pressure decreases.

Trace Contaminant Testing. The full-sized module cell (as opposed to the single cell used in the other parametric tests) was selected to determine the effect of trace contaminants of 1%  $N_2H_4$  and 1%  $NH_3$  in the feed gas mixture on the actual cell materials that would be used in the second and third stages of the separator. The voltage versus time curves for the two tests are shown in Figures 12 and 13. Trace quantities of  $N_2H_4$  and  $NH_3$  had little effect on separator performance. In the  $N_2H_4$  test, the voltage remained constant over the test period. The rise in voltage over the first hour or so is characteristic of electrochemical cells of this type and was observed even when trace  $N_2H_4$  or  $NH_3$  was not present. When  $NH_3$  was added to the feed gas, the voltage increased slightly during the test period but not enough to indicate  $NH_3$  electrode poisoning.

Baseline Operating Conditions. Based on the results of the Single-Cell Parametric Test Program, the baseline operating conditions for the second and third stage electrochemical  $N_2/H_2$  separator for use in a  $7.88 \times 10^{-5} \text{ kg/s}$  (15 lb/day)  $N_2$  generator detailed in Table 6.

### Second Stage Testing

A three-cell module representing the second stage of an electrochemical  $N_2/H_2$  separator was tested to insure the stage met the design specifications established through the Parametric Test Program (Table 6). The test program included a shakedown test, a design verification test and a 200 hour endurance test.

Shakedown Testing. During the shakedown testing it was found that the module would not hold anode to cathode differential pressures greater than  $1.38 \times 10^4 \text{ N/m}^2$  (2 psid). The problem was solved by the insertion of ethylene propylene gaskets between each electrode frame and the electrolyte matrix to prevent gases from leaking around the edge of the matrix.



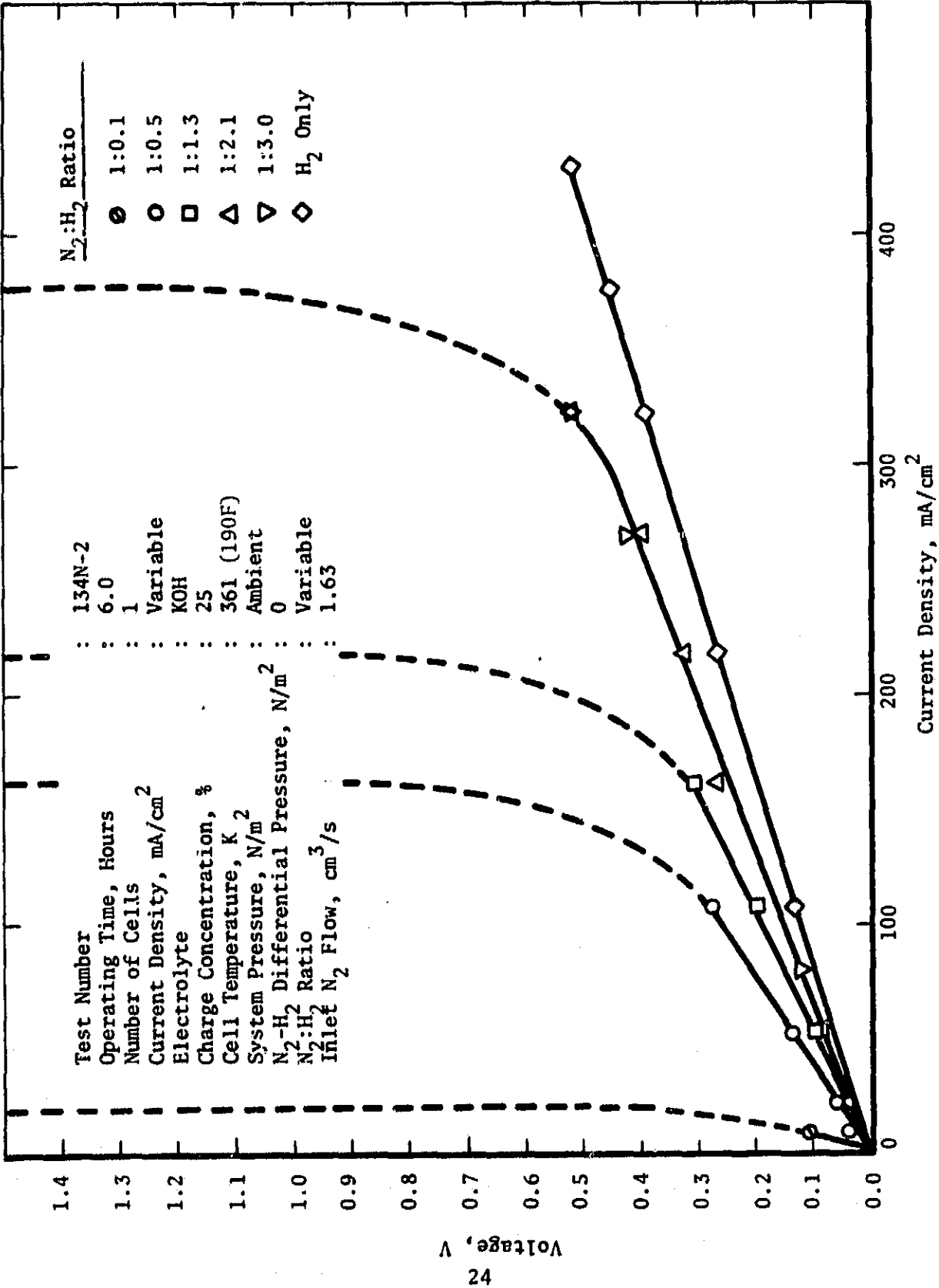


FIGURE 9 EFFECT OF N<sub>2</sub>:H<sub>2</sub> FEED RATIO ON CELL PERFORMANCE

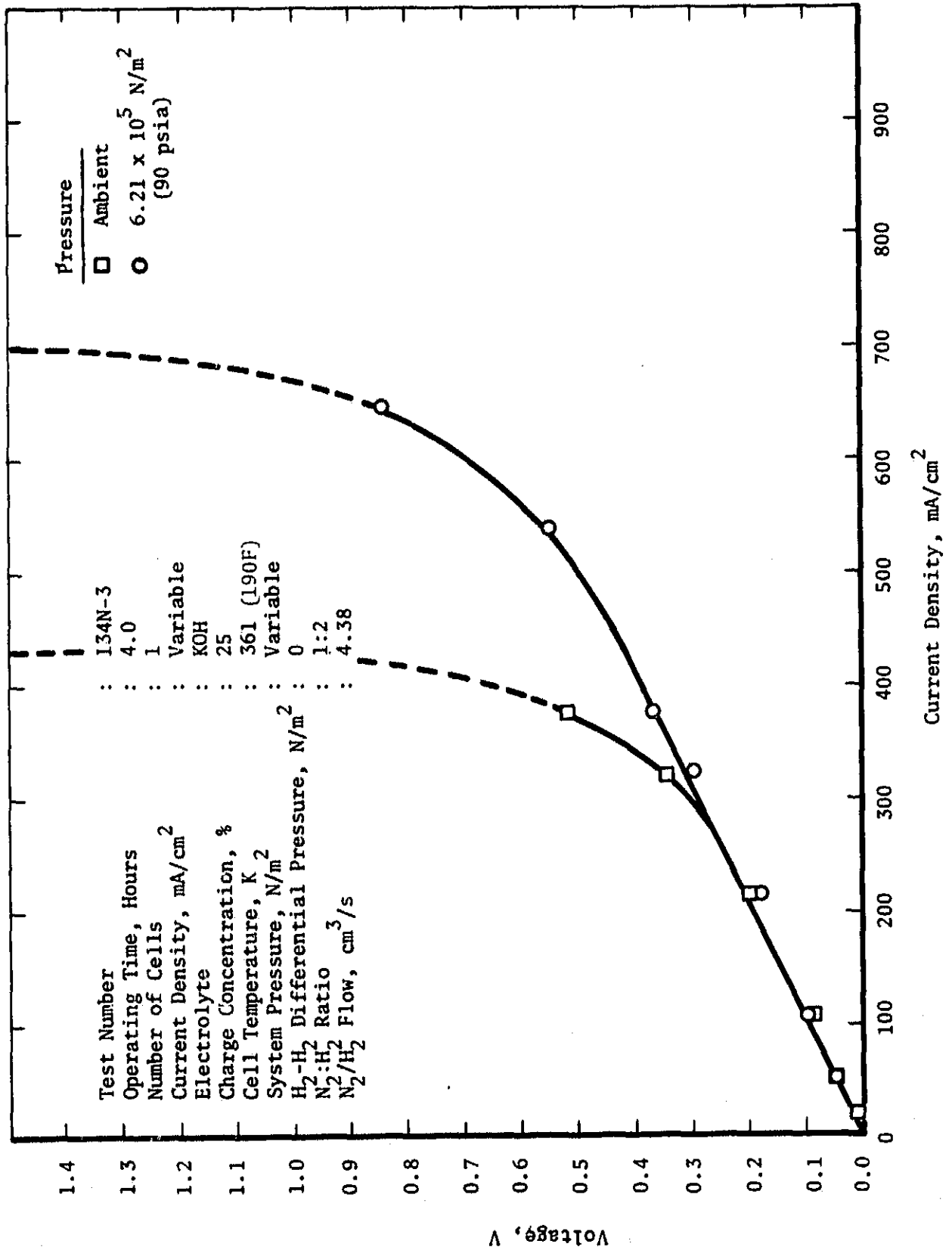


FIGURE 10 EFFECT OF PRESSURE ON CELL PERFORMANCE

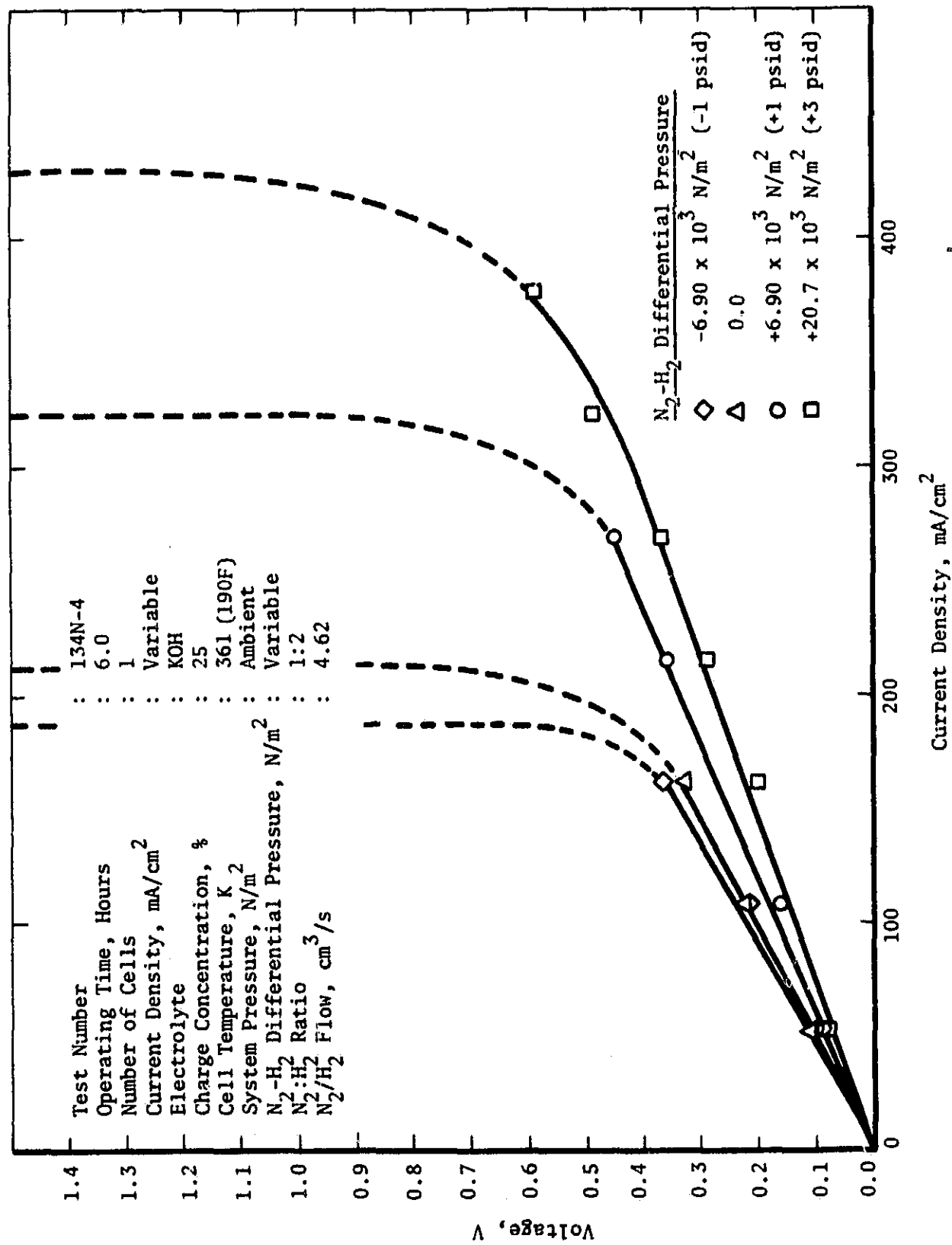


FIGURE 11 EFFECT OF DIFFERENTIAL PRESSURE ON CELL PERFORMANCE

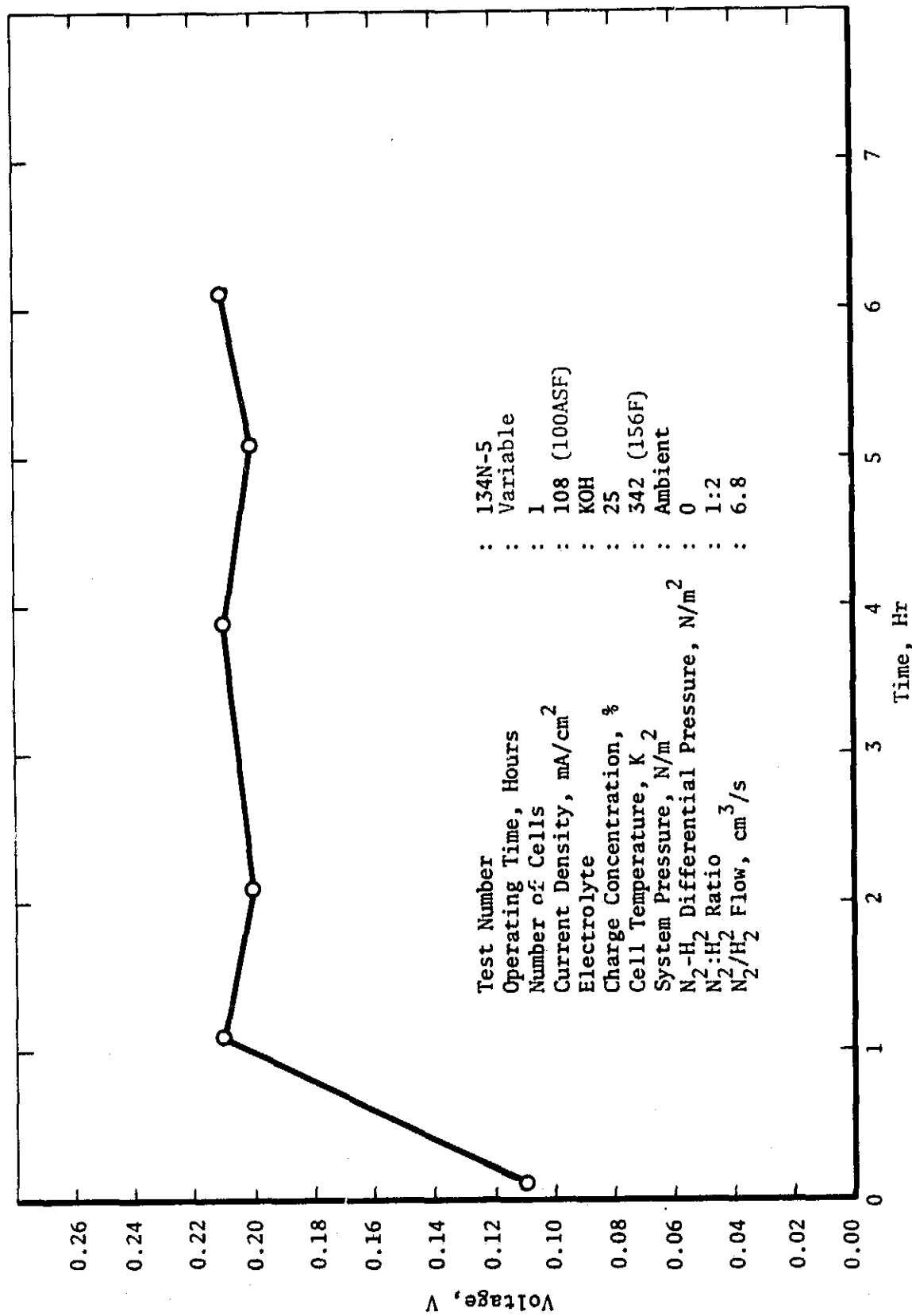


FIGURE 12 EFFECT OF 1% N<sub>2</sub>H<sub>4</sub> CONTAMINANT ON CELL PERFORMANCE

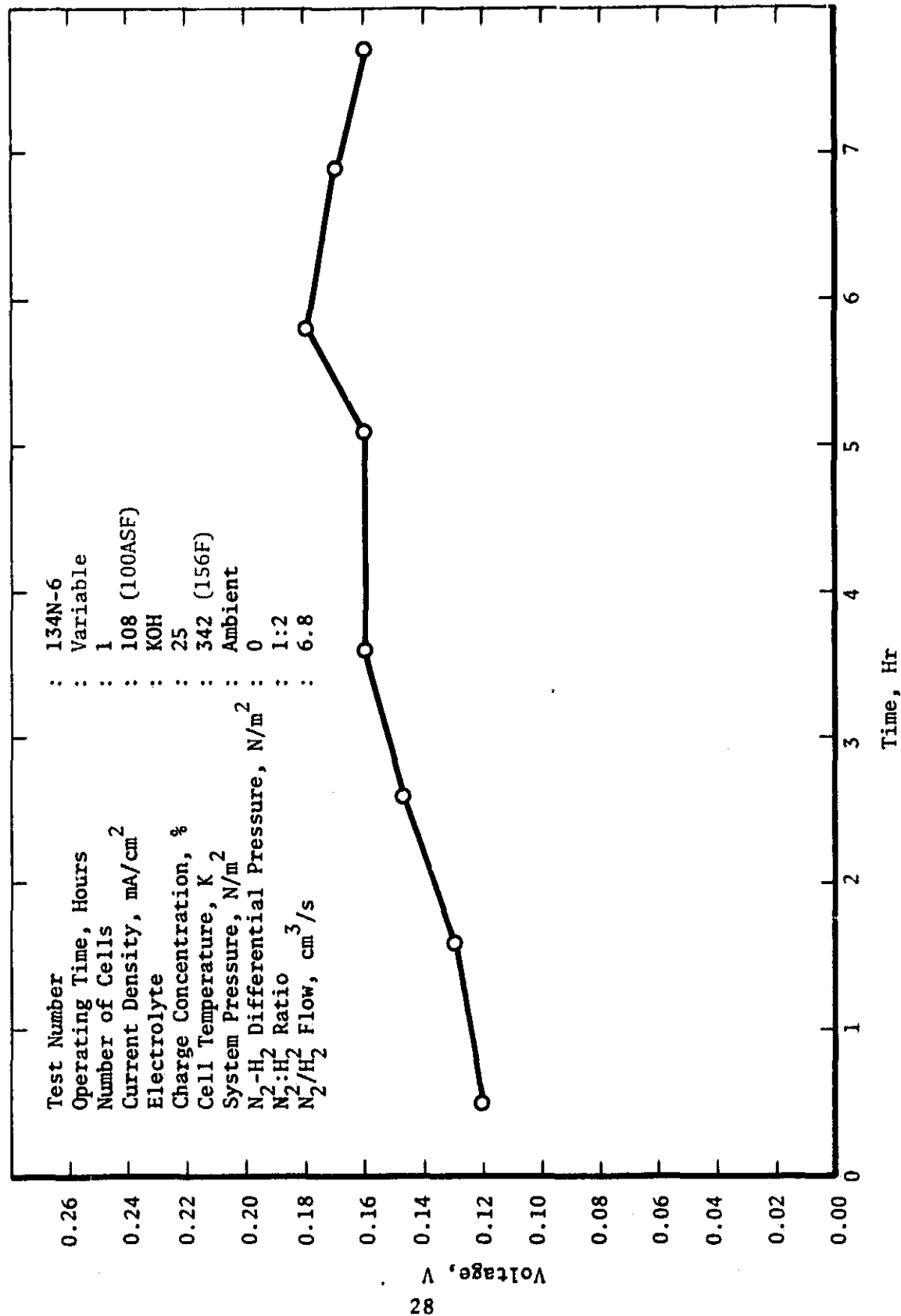


FIGURE 13 EFFECT OF 1% NH<sub>3</sub> CONTAMINANT ON CELL PERFORMANCE

TABLE 6 BASELINE OPERATING CONDITIONS

	Second Stage	Third Stage
$N_2/H_2$ Feed		
$N_2$ Flow, kg/s (lb/day)	$7.88 \times 10^{-5}$ (15)	$7.88 \times 10^{-5}$ (15)
$H_2$ Flow, kg/s (lb/day)	$1.12 \times 10^{-6}$ (0.21)	$1.12 \times 10^{-7}$ (0.02)
$H_2$ Concentration, % v/v	16.7	2.0
Temperature, K (F)	295 (72)	298 (77)
Pressure, N/m <sup>2</sup> (psia)	$6.34 \times 10^5$ (92)	$6.27 \times 10^5$ (91)
$N_2$ Product		
$N_2$ Flow, kg/s (lb/day)	$7.88 \times 10^{-5}$ (15)	$7.88 \times 10^{-5}$ (15)
$H_2$ Flow, kg/s (lb/day)	$1.12 \times 10^{-7}$ (0.02)	$1.12 \times 10^{-8}$ (0.002)
$H_2$ Concentration, % v/v	2.0	< 0.1
Temperature, K (F)	298 (77)	300 (80)
Pressure, N/m <sup>2</sup> (psia)	$6.27 \times 10^5$ (91)	$6.21 \times 10^5$ (90)
$N_2-H_2$ Differential Pressure, N/m <sup>2</sup> (psid)	$6.90 \times 10^3$ (1)	$6.90 \times 10^3$ (1)
$H_2$ Product		
$H_2$ Flow, kg/s (lb/day)	$1.01 \times 10^{-6}$ (0.19)	$1.01 \times 10^{-7}$ (0.019)
Temperature, K (F)	298 (77)	300 (80)
Pressure, N/m <sup>2</sup> (psia)	$6.21 \times 10^5$ (90)	$6.14 \times 10^5$ (89)
Module		
Number of Cells	3	1
Current per cell, A	32.2	9.7
Electrode area, cm <sup>2</sup> (ft <sup>2</sup> )	223 (0.24)	223 (0.24)
Current density, mA/cm <sup>2</sup> (ASF)	144 (134)	43.5 (40.4)
Voltage per cell, V	0.37	1.00
Temperature, K (F)	298 (77)	300 (80)
Electrolyte	KOH	KOH
Charge concentration, % w/w	25	25

Design Verification Testing. Figure 14 shows the individual cell voltages of the second stage module as a function of time for baseline operating conditions. The second stage was run at a constant current density,  $144 \text{ mA/cm}^2$  (134 ASF) and the voltage of each cell was lower than the design voltage of 0.37 V.

Endurance Testing. The endurance test consisted of running the second stage module continuously without shutdown for 200 hours at baseline operating conditions. Figure 15 shows the individual cell voltages as a function of time. During the testing two out-of-balance moisture conditions were made to occur (approximately 35 and 150 hours into the testing) causing cell voltage to rise. The first moisture imbalance corresponded to dry conditions, i.e.,  $\text{H}_2\text{O}$  was removed from the electrolyte causing the electrolyte concentration to increase. The second imbalance corresponded to wet conditions, i.e.,  $\text{H}_2\text{O}$  was deposited in the electrolyte causing the electrolyte concentration to decrease. As a result, the moisture tolerance of the module was established to be the dew point above the electrolyte  $\pm 1\text{K}$  ( $\pm 2\text{F}$ ).

### Third Stage Testing

A single-cell module was tested representing the third stage of an electrochemical  $\text{N}_2/\text{H}_2$  separator. The test program included a shakedown test and a 60 hour design verification test.

Shakedown Testing. During the shakedown test the third stage performed its function properly. As a result no changes were made to the module design.

Design Verification Testing. Figure 16 shows cell current as a function of time for baseline operating conditions. The third stage was run at a constant voltage (1.00 V) and the current was allowed to vary causing the corresponding change in the amount of  $\text{H}_2$  removed. The decrease in cell current at 12 hours corresponded to a decrease in the  $\text{H}_2$  flow that was added to the  $\text{N}_2$  stream by the test facility to simulate the  $\text{N}_2/\text{H}_2$  feed to the third stage. The current increased when the  $\text{H}_2$  flow was returned to the design level.

### ALTERNATE TECHNIQUES FOR $\text{N}_2/\text{H}_2$ SEPARATION

The separation of gaseous  $\text{N}_2$  and  $\text{H}_2$  mixtures can be accomplished by a variety of processes which take advantage of the difference in any specific property exhibited by either the  $\text{N}_2$  or the  $\text{H}_2$ . Such properties include vapor pressure, solubility, affinity to adsorption, diffusion coefficients, molecular weight, density and chemical reactivity. Based on a literature survey, the following methods of separating  $\text{N}_2/\text{H}_2$  mixtures were identified:

1. Heated palladium-silver membranes.
2. Organic polymer membranes.
3. Cryogenic separation.

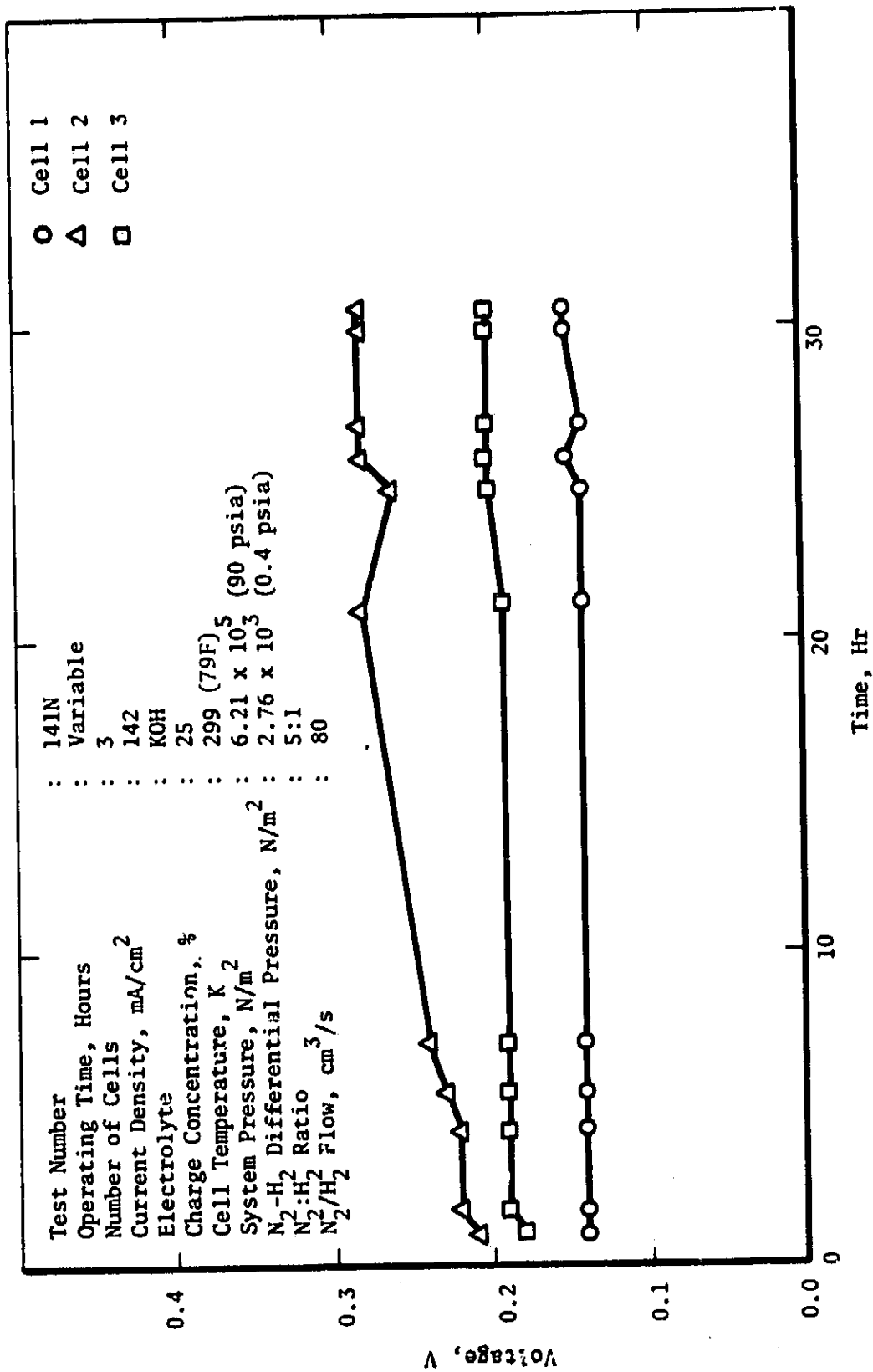


FIGURE 14 SECOND-STAGE DESIGN VERIFICATION TEST



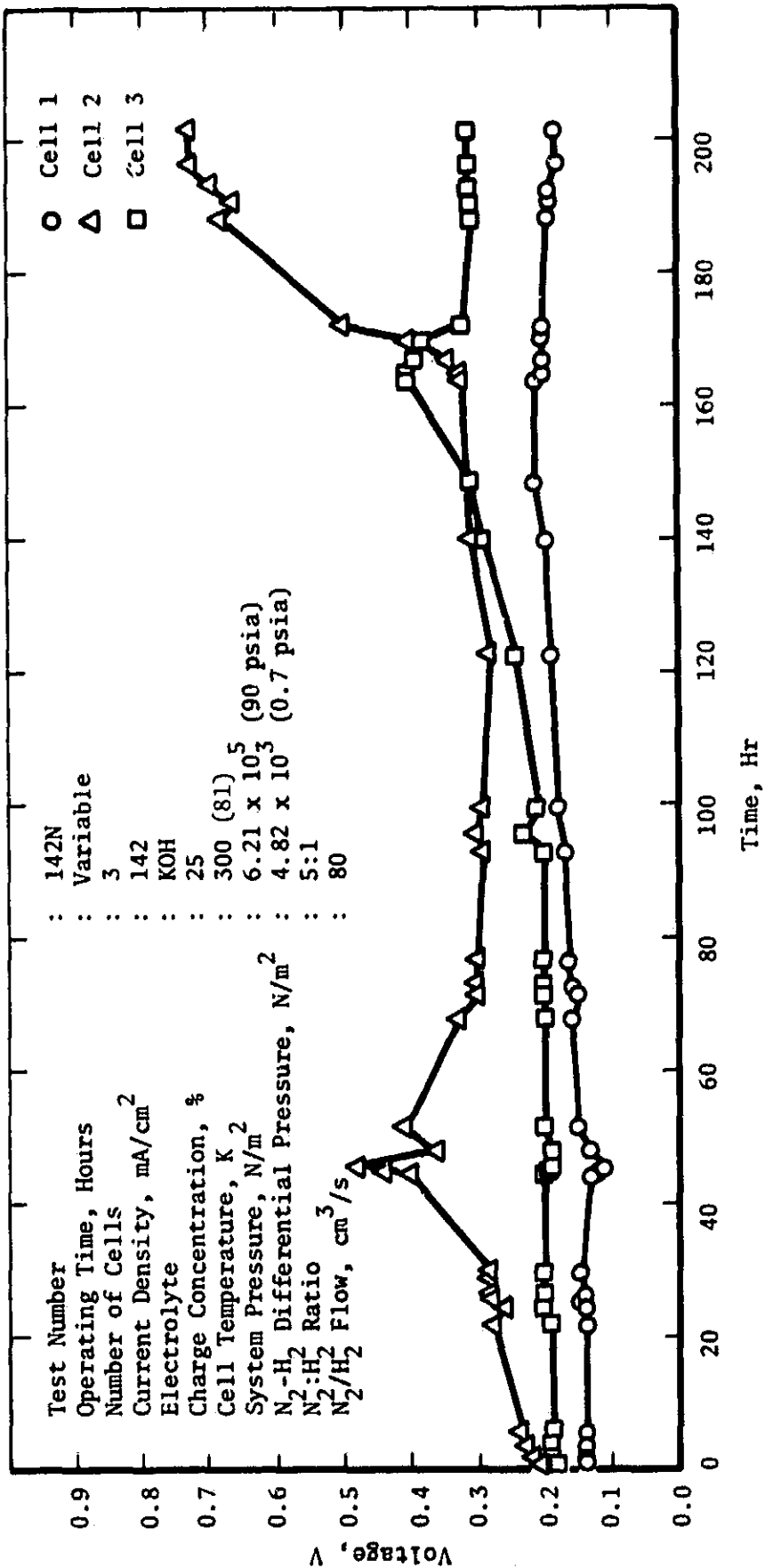


FIGURE 15 SECOND-STAGE ENDURANCE TEST

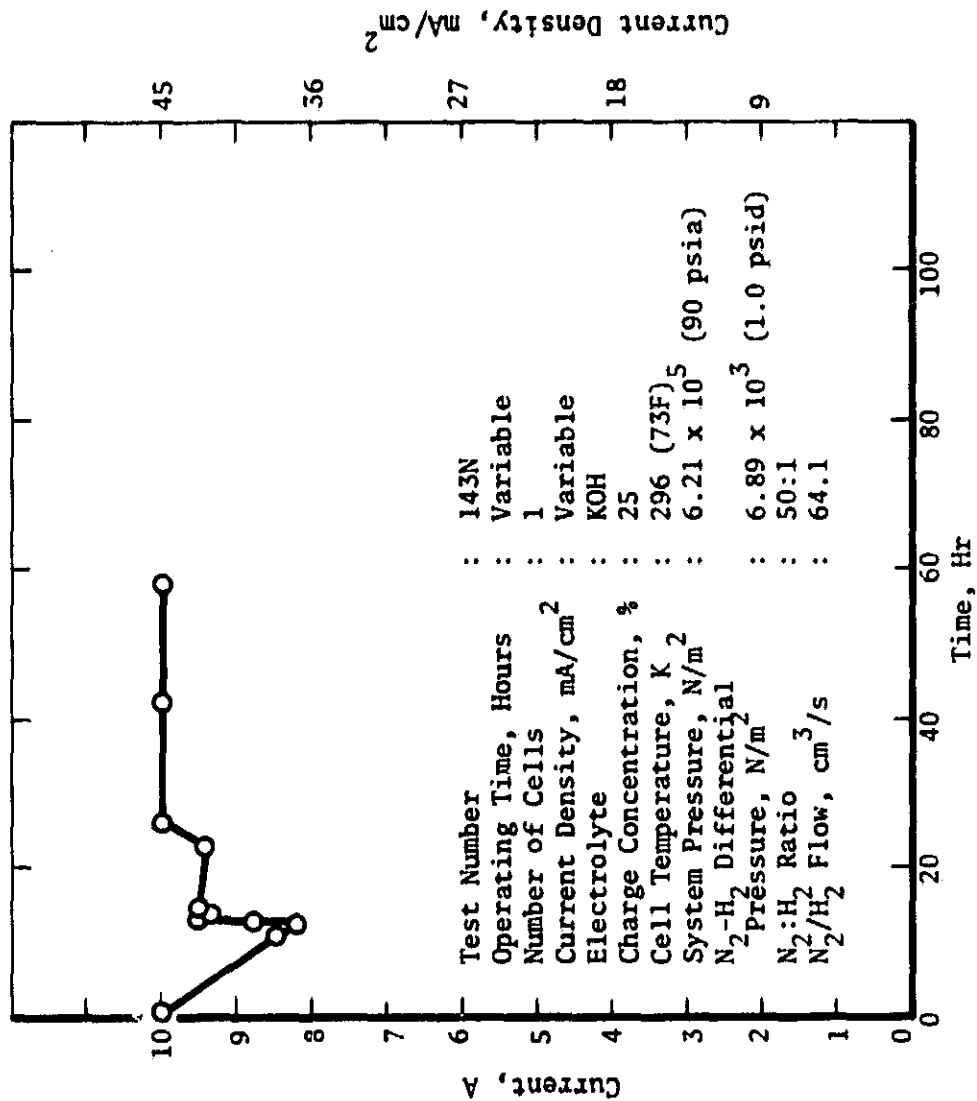


FIGURE 16 THIRD-STAGE DESIGN VERIFICATION TEST

4. Removal by a  $H_2$  reaction.
5. Selective adsorption by a solid.
6. Gas centrifuge.
7.  $H_2$  removal as a hydride.
8. Parametric Pumping.

#### Heated Palladium-Silver Membrane<sup>(3)</sup>

An alloy of 75% palladium (Pd) and 25% silver (Ag) can absorb as much as 800 to 900 times its own volume of  $H_2$  under certain temperature and pressure conditions. In addition,  $H_2$  is very mobile within the alloy so that it can travel from one surface of the membrane to the other under the driving force of a  $H_2$  partial pressure difference. The diffusivity of  $H_2$  through the Pd/Ag alloy is  $6.2 \times 10^{-4} \text{ cm}^2/\text{s}$  at 644K (700F) and less than  $2.6 \times 10^{-10} \text{ cm}^2/\text{s}$  for all other substances diffusing through Pd/Ag. The very high selectivity of Pd/Ag for  $H_2$  allows a highly efficient separation of  $H_2$  from other gases.

Diffusion Unit. The Pd/Ag membrane can be formed into thin tubes which are supported on the inside by a stainless steel wire helix. By closing the membrane tubes at one end and matching the other ends to a manifold, a Pd/Ag diffusion unit resembling a small shell and tube heat exchanger is formed. The Pd/Ag diffusion unit is shown in Figure 17.

Operation. Figure 18 is the  $N_2/H_2$  separator subsystem schematic based on the Pd/Ag diffusion unit. The diffusion unit consists of two stages with the gases manifolded internally by baffles. The  $N_2/H_2$  feed mixture enters the diffusion unit in the shell side of the first stage at  $1.03 \times 10^6 \text{ N/m}^2$  (150 psia) and 644K (700F). Hydrogen diffuses into the tubes under a  $H_2$  partial pressure driving force and is collected at  $1.72 \times 10^5 \text{ N/m}^2$  (25 psia) for use. The  $H_2$ -depleted mixture from the shell side of the first stage enters the shell side of the second stage. The remaining  $H_2$  diffuses into the tubes of the second stage and is vented to space vacuum. The purified  $N_2$  from the shell side of the second stage, containing less than 0.1%  $H_2$ , is delivered at approximately the same pressure as the  $N_2/H_2$  feed.

#### System Advantages.

1. The quality of separation attainable with the Pd/Ag diffusion unit is exceptionally good. The recovered  $H_2$  contains less than 10 ppb impurities because of the great difference in relative permeation rates of  $N_2$  and  $H_2$  through the membrane.
2. The Pd/Ag diffusion unit is capable of operating efficiently over a broad operating temperature range, 589K to 700K (600F to 800F). The system, therefore, does not require precise temperature regulation.

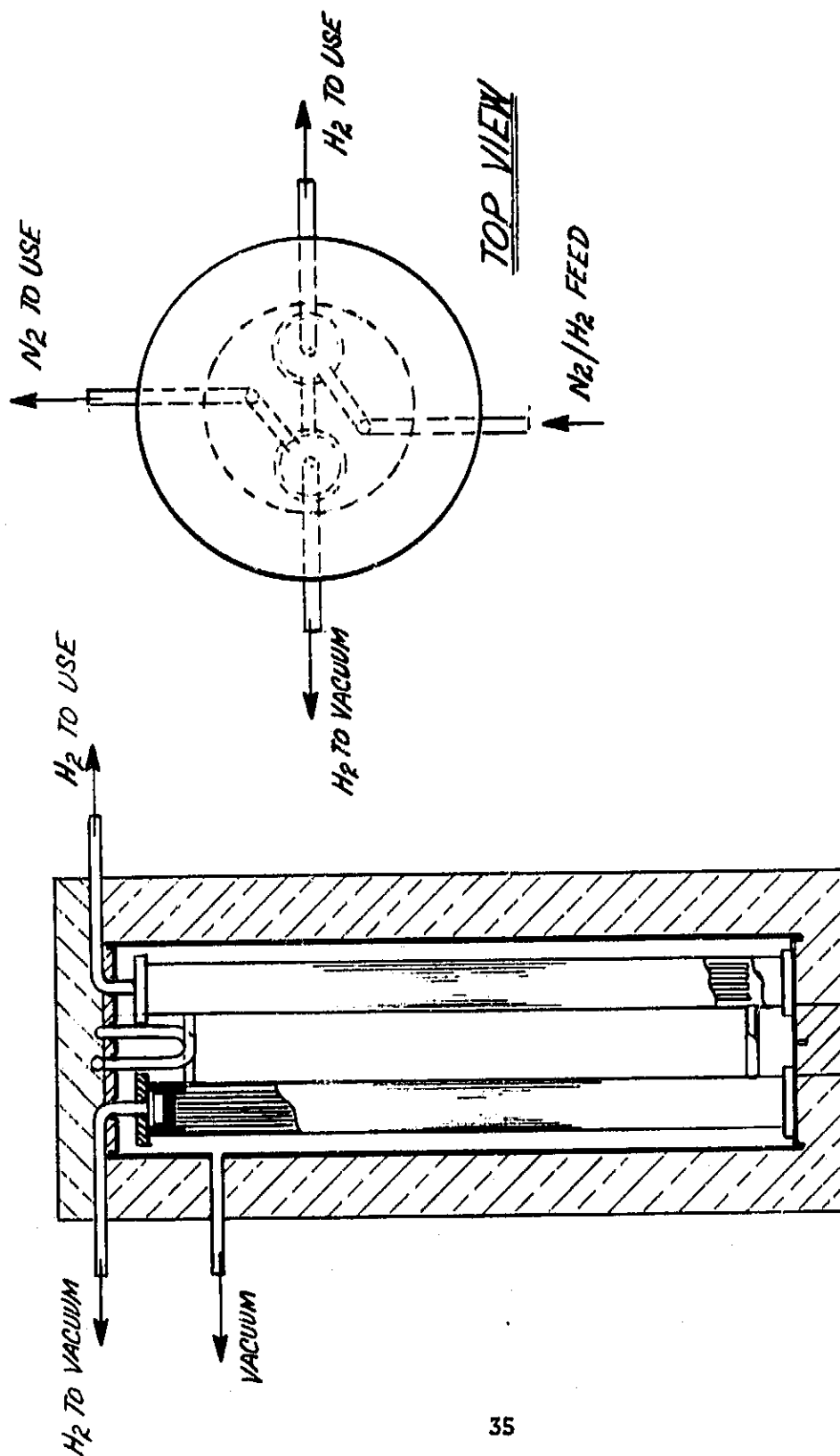


FIGURE 17 PALLADIUM-SILVER DIFFUSION UNIT

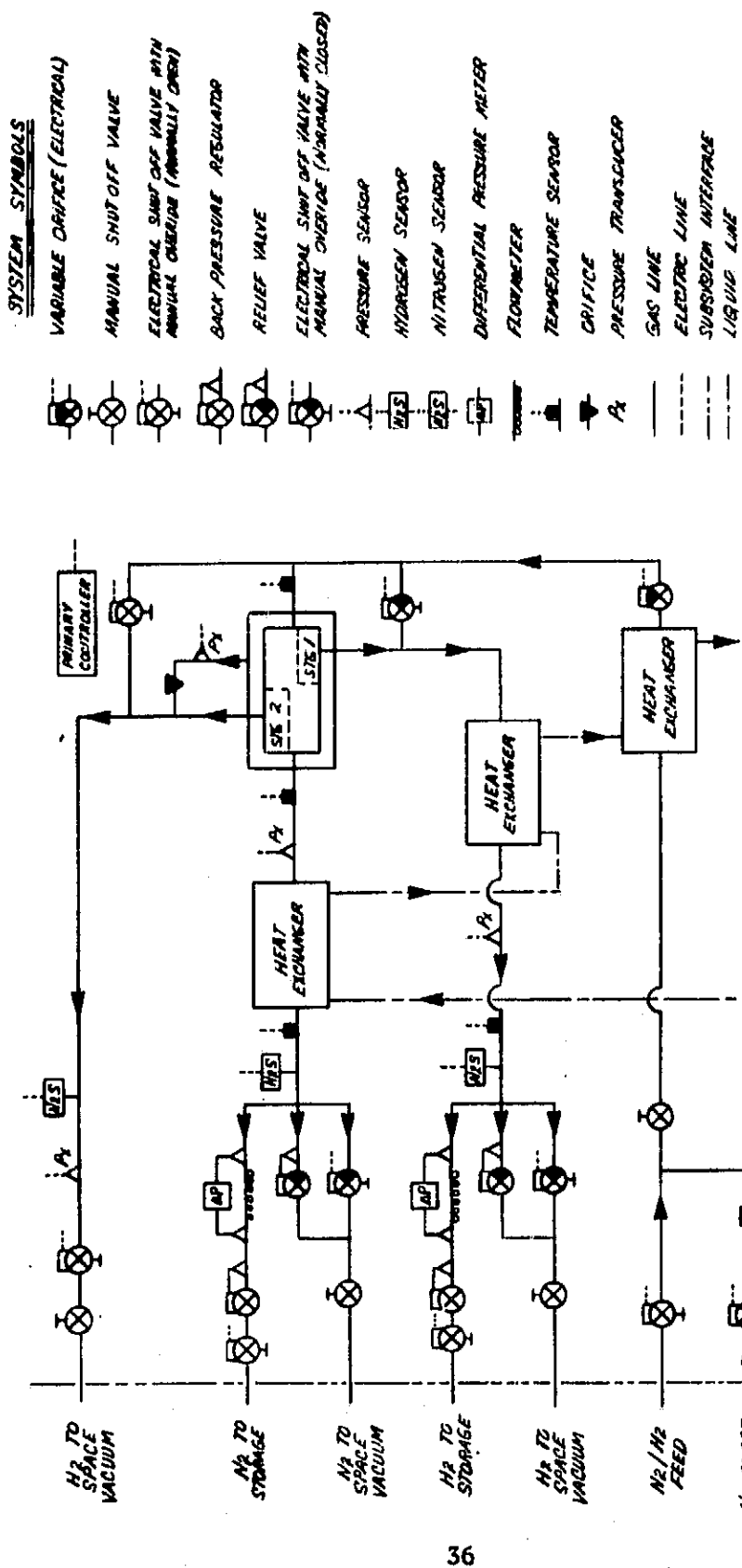


FIGURE 18 PALLADIUM-SILVER  $N_2/H_2$  SEPARATOR SUBSYSTEM SCHEMATIC

3. The reliability characteristics of the Pd/Ag diffusion unit are similar to those of a shell and tube heat exchanger. All parts of the diffusion unit are either welded or sweated in place and, therefore, have a very high reliability factor.
4. The Pd/Ag membrane and other parts of the diffusion unit, which are in contact with the  $N_2/H_2$  feed stream, are insensitive to trace quantities of  $NH_3$  and  $N_2H_4$  which are present in the dissociation of  $N_2H_4$ . Also, since Pd is a catalyst for the dissociation of  $NH_3$  and  $N_2H_4$ , the Pd/Ag diffusion unit itself serves to dissociate some of the  $NH_3$  and  $N_2H_4$  present in the  $N_2/H_2$  feed.
5. Because of the extremely high  $H_2$  permeability of the Pd/Ag membrane, only 1059 and 1310  $cm^2$  (1.14 and 1.41  $ft^2$ ) of diffusion surface area are required in the first and second stages, respectively, to meet the design requirements of  $7.88 \times 10^{-5}$  kg/s (15 lb/day) of  $N_2$ . The diffusion unit would weigh only 3.6-4.1 kg (8-9 lb).

#### System Disadvantages.

1. The Pd/Ag diffusion unit must operate at elevated temperatures resulting in higher heat losses, higher stress on valves, pressure sensors, etc., and a loss of  $H_2$  through the metal container holding the Pd/Ag diffusion tubes ( $H_2$  is slightly permeable in other metals at elevated temperatures).
2. Cyclic operation of the diffusion unit decreases the reliability of the welded and sweated joints and hence decreases the overall reliability of the diffusion unit.
3. The driving force in the diffusion unit is the difference in  $H_2$  partial pressures across the membrane. When the final concentration of  $H_2$  in the product  $N_2$  is reduced to very low levels, the pressure on the opposite side of the membrane must be approximately a vacuum. The  $H_2$  in the second stage then, is lost to space vacuum. Should the loss of  $H_2$  be critical, or if it is undesirable to vent  $H_2$  to space, it would be necessary to recompress the  $H_2$  or recover it electrochemically or by other means.
4. Since Pd/Ag catalyzes the reaction of  $H_2$  and oxygen ( $O_2$ ),  $O_2$  must be excluded from the system. Should  $O_2$  be allowed to enter the system in the presence of  $H_2$ , the highly exothermic reaction of  $H_2$  and  $O_2$  would crack the Pd/Ag tubes rendering the unit inoperable. The system, therefore, must incorporate  $N_2$  purge capabilities to insure that  $O_2$  is removed from the diffusion unit prior to the introduction of  $H_2$ .

### Organic Polymer Membrane<sup>(4)</sup>

The transmission of fluids through polymeric membranes is known as selective solubility diffusion. The differential rate of permeation of each gas through the polymer membrane is proportional to the partial pressure difference across the membrane. The constant of proportionality is called the permeability coefficient. The separation (or selectivity) factor is defined as the ratio of the permeability coefficients of the gases involved. The separation factor for  $H_2$  to  $N_2$  through hollow polyester fibers at 322K (120F) is 42.3.

Diffusion Unit. Hollow polyester fibers can be arranged into a shell and tube diffusion unit resembling a shell and tube heat exchanger. The polymer diffusion unit is shown in Figure 19.

Operation. Figure 20 is the  $N_2/H_2$  separator subsystem schematic based on the polymer diffusion unit. As in the Pd/Ag method, the separation is carried out in two stages. The  $N_2/H_2$  feed mixture at  $1.03 \times 10^6$  N/m<sup>2</sup> (150 psia) and 322K (120F) flows through the inside of the hollow fibers. In the first stage, 80% of the  $H_2$  permeates through the membrane, with a small amount of  $N_2$  (4-5%), and is recovered on the shell side at  $1.72 \times 10^5$  N/m<sup>2</sup> (25 psia). In the second stage the shell side is connected to space vacuum where the  $H_2$  and  $N_2$  that diffuse through the membrane are lost to space vacuum. In order to obtain the desired  $N_2$  product purity (99.9%), however, a further 10-15% loss of the feed  $N_2$  is incurred in the second stage.

#### System Advantages.

1. The weight and volume of the polymer gas permeator are comparable to the Pd/Ag diffusion unit.
2. The permeator does not operate at elevated temperatures.
3. Since the polymer membrane does not catalyze the reaction of  $O_2$  and  $H_2$ , a  $N_2$  purge is not required to protect the diffusion unit. (a)
4. The reliability of the permeator unit is approximately the same as that of the Pd/Ag diffusion unit.
5. The separation of gases with polymer membrane fibers is a relatively new technique. Many new and better suited polymers are being developed to improve currently available designs.

#### System Disadvantages.

1. The life of the permeator unit is decreased by trace quantities of  $NH_3$  and  $N_2H_4$ .

(a) Normal safety procedures, however, require a  $N_2$  purge for all lines and components containing  $H_2$ .

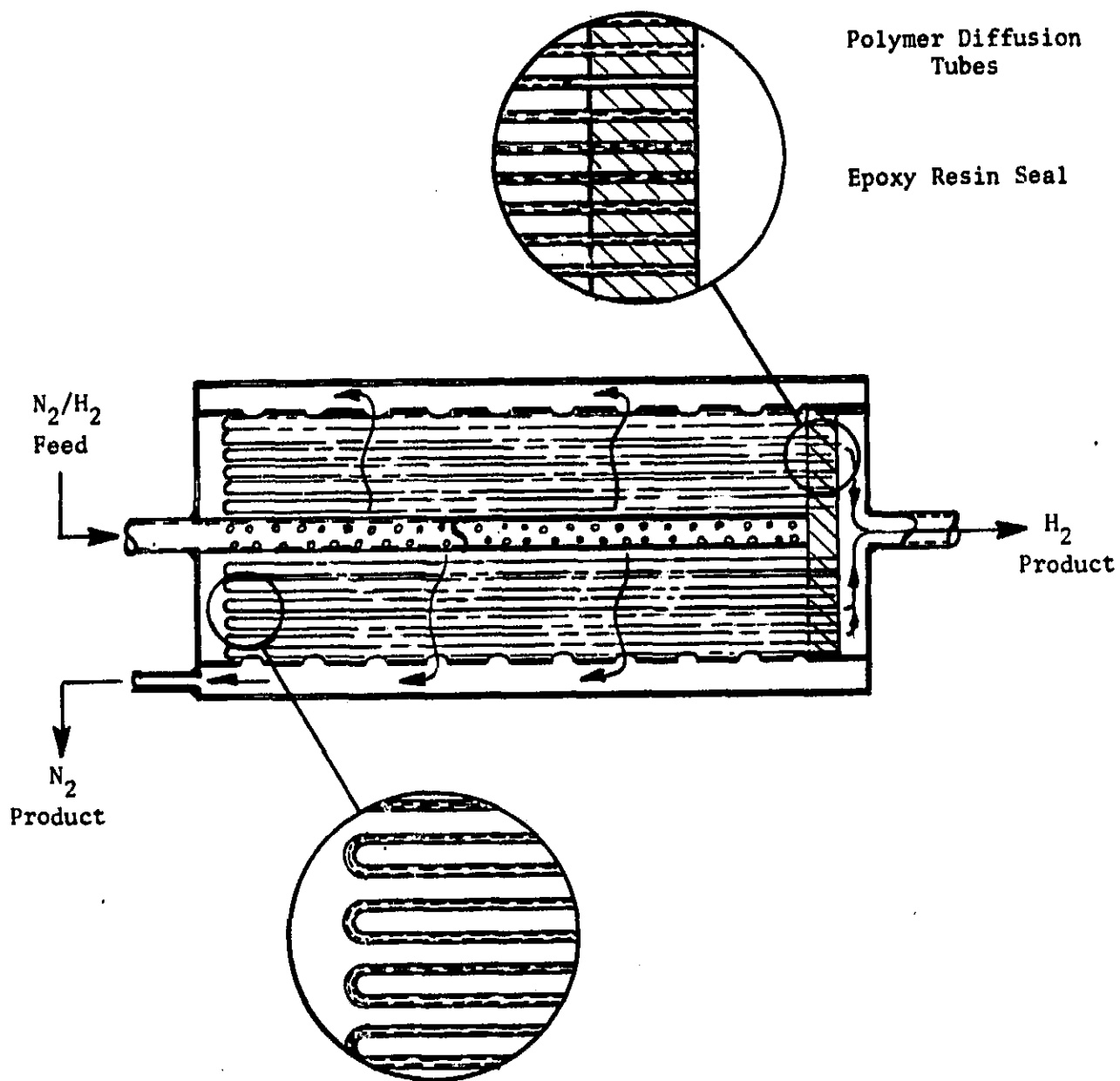


FIGURE 19 SINGLE STAGE POLYMER MEMBRANE DIFFUSION UNIT



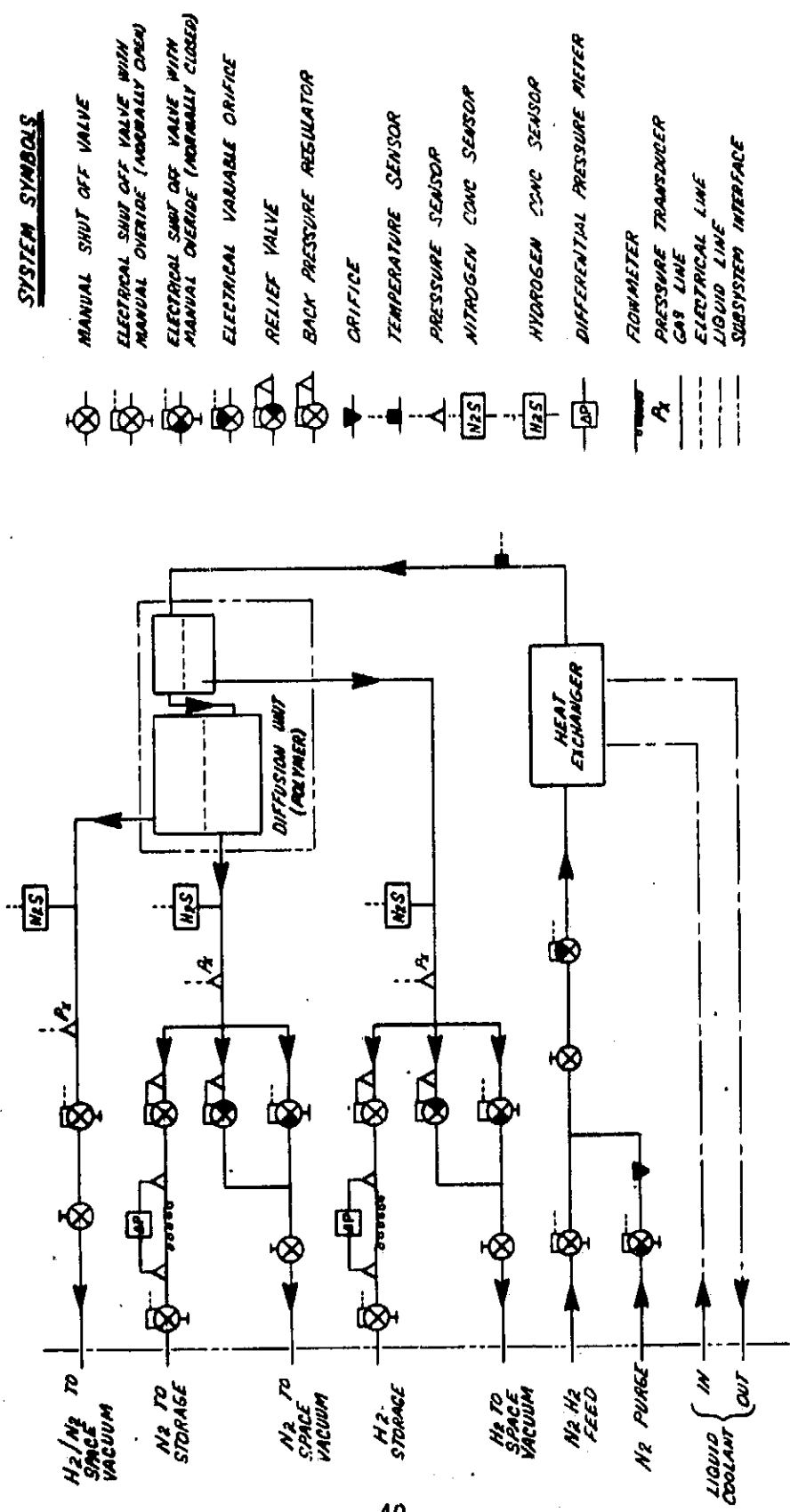


FIGURE 20 POLYMER N<sub>2</sub>/H<sub>2</sub> SEPARATOR SUBSYSTEM SCHEMATIC

2. Permeator life decreases as temperature increases above 322K (120F).
3. Approximately 4-5% and 10-15% of the feed  $N_2$  is lost in the first and second stages, respectively. The feed gas, then, must contain more than  $7.88 \times 10^{-5}$  kg/s (15 lb/day) of  $N_2$  to account for the losses.
4. The container which supports the polymer tubes and manifolds the gases is not presently designed for lowest weight. A low-weight container would have to be developed for spacecraft applications.

### Cryogenic Separation

The separation of gaseous mixtures of  $N_2/H_2$  can be achieved by cooling the mixture below the critical temperature of  $N_2$  so that most of the  $N_2$  condenses. Hydrogen would remain in the vapor phase since  $H_2$  condenses at an even lower temperature. The process is completed by separating the liquid ( $N_2$ ) and vapor ( $H_2$ ) phases.

Operation. Figure 21 is the schematic of the cryogenic separation process. The highly compressed feed gas mixture would initially be cooled in a regenerative heat exchanger by evaporating the already separated  $N_2$  condensate and heating the cold separated gaseous  $H_2$ . The compressed gases are further cooled to the critical temperature of  $N_2$ , 126K (-233F) by an isentropic expansion in a turbo-expander. Further reduction in system temperature required to condense  $N_2$  is accomplished by an adiabatic expansion through a Joule-Thompson valve. The mean Joule-Thompson coefficient for such an expansion was found to be  $2.13 \times 10^{-4}$  K<sup>2</sup>/N (0.21 K/atm). (a)

The pressure drop through a Joule-Thompson valve to achieve the required separation is approximately  $3.04 \times 10^7$  N/m<sup>2</sup> (300 atm). The pressure drop that occurred in the turbo-expander to bring the feed gas to 126K (-233F) was approximately  $1.01 \times 10^7$  N/m<sup>2</sup> (100 atm). Hence, the total pressure to which the gas mixture must be compressed is  $4.05 \times 10^7$  N/m<sup>2</sup> (400 atm).

System Advantages. The system was found to have no advantages over other methods considered for  $N_2/H_2$  separation.

### System Disadvantages.

1. System weight is high because of bulky equipment (compressor and expansion engine).
2. Excessive system power is required to compress the gases for expansion.

(a) Extrapolated from handbook values, the error could possibly be as high as 20%.

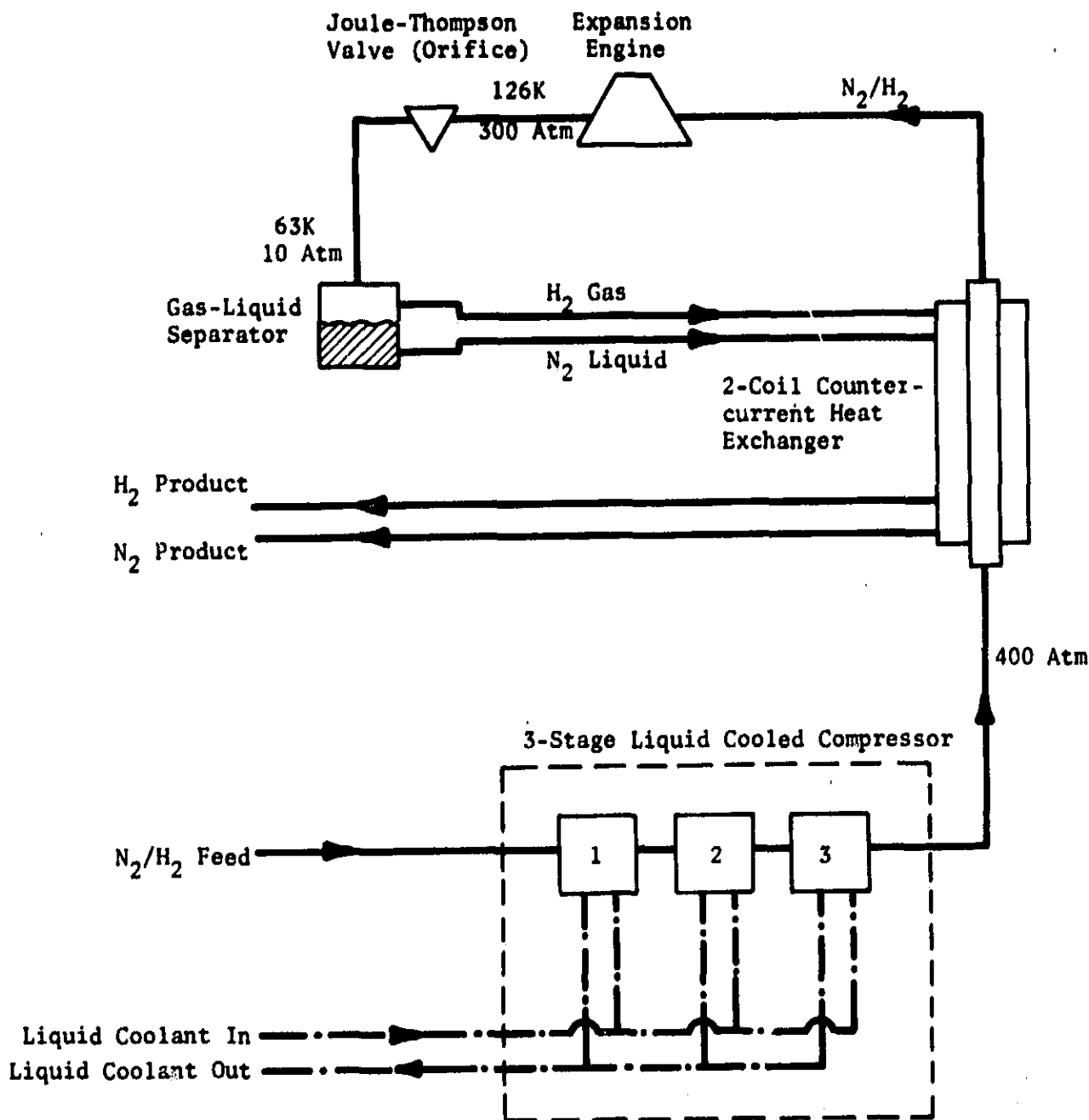


FIGURE 21 CRYOGENIC  $N_2/H_2$  SEPARATION PROCESS SCHEMATIC

3. The compressor and expansion engine have a high noise level.
4. The cryogenic  $N_2/H_2$  gas mixture would present a materials problem.

#### Removal By Reaction

The  $H_2$  component of the  $N_2/H_2$  mixture can be removed by reaction with a stoichiometric amount of  $O_2$  to form  $H_2O$  and the reaction mixture is then dried by passing the product stream over an alumina bed.

#### System Advantages.

1. The process is simple and can be easily regulated.
2. The power requirements to heat the mixture for reaction are low.
3. The process would integrate well as the final stage for another process by using an excess of  $O_2$  to remove the last traces of  $H_2$ .

#### System Disadvantages.

1. There is a high equivalent weight penalty for using  $O_2$  aboard spacecraft.
2. Mixing the exact stoichiometric amounts of  $O_2$  and  $H_2$  would be difficult. (If the reaction took place in an excess of  $O_2$ , the  $N_2$  product stream could only be used for air make-up).
3. The reaction of  $H_2$  and  $O_2$  is highly exothermic, thus there is a high heat rejection penalty.
4. The  $H_2$  is lost for direct use from the  $N_2/H_2$  separator.

#### Selective Adsorption by a Solid<sup>(5)</sup>

Gaseous  $N_2/H_2$  mixtures can be separated using molecular sieves or certain metal chelates which adsorb  $N_2$  or  $H_2$  selectively from the mixture. The separation is accomplished using a cyclic adsorption process.

The processing technique, called heatless fractionation, uses two adsorption beds operated in parallel alternation. In the first bed either  $H_2$  or  $N_2$  from the pressurized gas stream is removed by the adsorbent bed. When the bed becomes saturated with the removed component, the flow is switched to the second adsorbent bed where the adsorption process continues. The first bed is then depressurized releasing the adsorbed gas for collection. The process continues with the two beds alternating in function.

Since the technique is relatively new (1963) a great deal of development remains, including identifying new and better adsorption materials.

### Gas Centrifuge

When a vertical cylinder containing a gas mixture is rotated about its axis at a high angular velocity, the gaseous mixture will tend to separate with the high molecular weight component concentration near the wall of the cylinder and low molecular weight component concentration towards the axis of the cylinder. The quality of the separation depends on the difference in the molecular weights of the two constituents. Since  $H_2$  has a relatively low molecular weight compared with  $N_2$ , a high separation is possible. The separation of gas mixtures with a gas centrifuge has not been extensively used on a laboratory scale since much better results have been obtained using traditional separation techniques.

### $H_2$ Removal as a Hydride<sup>(6)</sup>

Hydrogen can be removed by adsorption from a  $N_2/H_2$  mixture and stored in the form of hydrides. Hydrides have the general formula  $AB_5H_x$  in which A represents Lanthanide (La, also known as a rare earth), B is nickel (Ni) or cobalt (Co), and H is hydrogen. As the  $H_2$  gas mixture comes in contact with the  $H_2$  binder ( $LaNi_5$ ), the  $H_2$  adsorbs forming a hydride. The amount of  $H_2$  adsorbed depends on system temperature and pressure. The  $H_2$ , however, can be effectively adsorbed and desorbed at room temperature and pressures of just a few atmospheres.

The removal of  $H_2$  as a hydride is similar to the selective adsorption process previously discussed. The major advantages of the hydride formation are the low pressure and temperature required for separation.

### Parametric Pumping

Parametric pumping is a relatively new separation technique that takes advantage of any specific property exhibited by one component of the mixture. The parametric pump, as discussed here, was used in conjunction with the  $H_2$  removal as a hydride process discussed above.

Operation. The parametric pumping process is shown in Figure 22. The adsorption unit consists of a long horizontal column with a gas reservoir at each end. The column bottom is packed with the  $H_2$  binder ( $LaNi_5$ ). The column is filled with the  $N_2/H_2$  mixture and pressurized such that the  $H_2$  is removed from the gas phase as a hydride. When equilibrium is reached, the  $N_2$ -rich gas is forced out of the column with more pressurized  $N_2/H_2$  feed gas. Hydrogen-rich gas from the  $H_2$  gas reservoir then forces the  $H_2$ -depleted  $N_2/H_2$  mixture into the  $N_2$  reservoir. The column is depressurized allowing the  $H_2$  to desorb. When equilibrium is reached the  $H_2$ -rich gas is forced out of the column with  $N_2/H_2$  feed. Nitrogen-rich gas from the reservoir then forces the  $N_2/H_2$  mixture into the empty  $H_2$ -rich gas reservoir. The column is again pressurized and the process repeats itself.

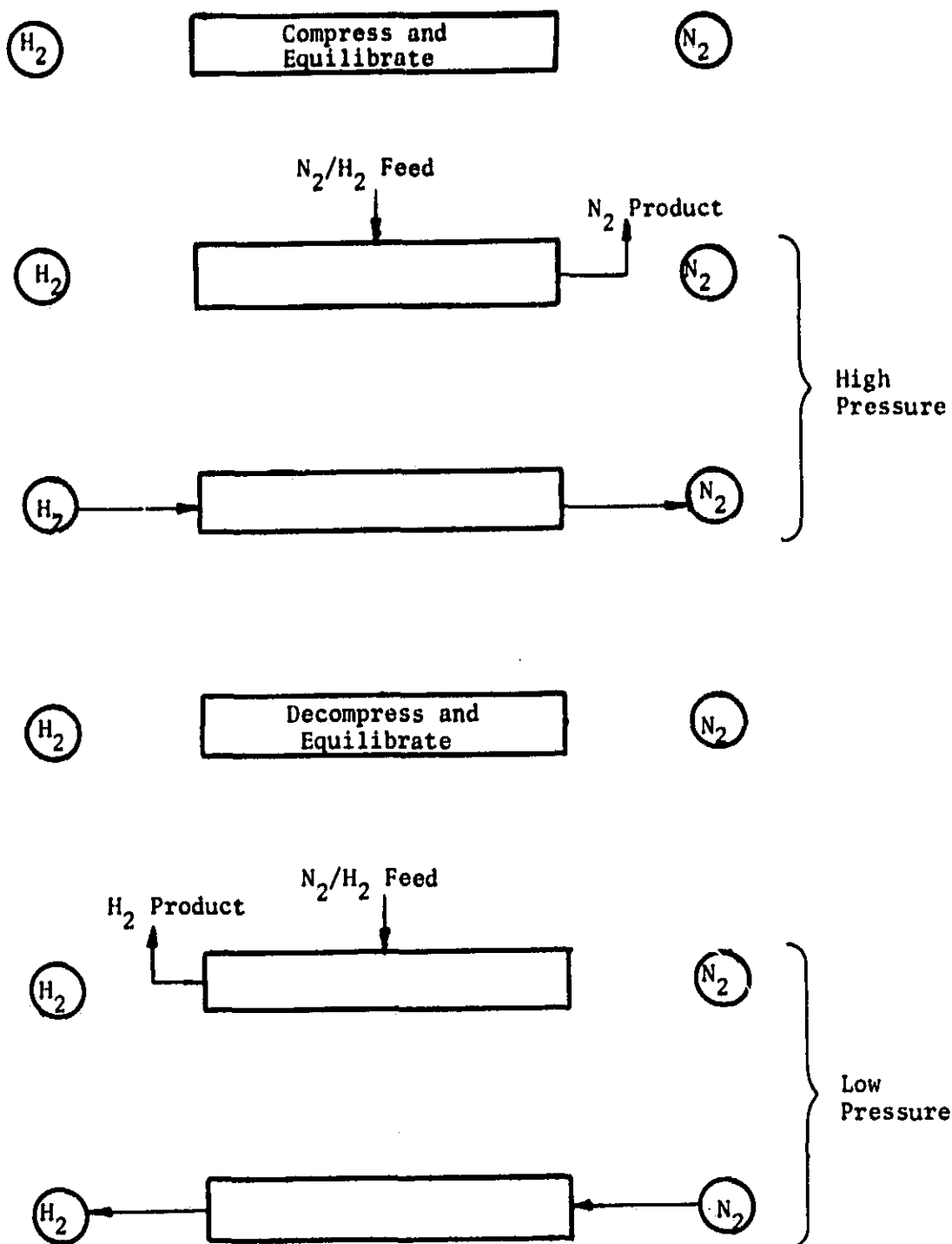


FIGURE 22 PARAMETRIC PUMPING PROCESS SCHEMATIC

System Advantages.

1. Parametric pumping can be used in conjunction with many separation methods.
2. High  $N_2$  purity is attainable.

System Disadvantages.

1. This technique requires excessive development time for spacecraft application.
2. It may require bulky, high weight equipment (compressors, pumps, etc.).

DESIGN RECOMMENDATIONS

Two cost-effective approaches to  $N_2/H_2$  separation were selected through a comparison study for use in a  $N_2$  Generator Subsystem. They are

1. the Pd/Ag diffusion unit, and
2. a combination first-stage organic polymer gas permeator and a second- and third-stage electrochemical  $N_2/H_2$  separator as shown in Figure 23.

A comparison of the Pd/Ag and polymer-electrochemical  $N_2/H_2$  separator is detailed in Table 7. A  $N_2/H_2$  separator system based on a Pd/Ag diffusion unit is estimated to have a total spared system equivalent weight of 317 kg (698 lb)<sup>(3)</sup>. The polymer-electrochemical system is estimated to have a total spared system equivalent weight of 257 kg (566 lb).

Comparison of Pd/Ag and Polymer-Electrochemical Approaches

Differences in system equivalent weights for the Pd/Ag and the polymer-electrochemical separator are due to the following dissimilarities in the two systems.

1. The polymer-electrochemical system operates at ambient temperatures, 322K (120F) or less, hence all heat removal takes place in one heat exchanger. The net savings are the two heat exchangers on the  $N_2$  and  $H_2$  product streams required by the Pd/Ag system.
2. The electrochemical third stage operates at constant voltage with the current reflecting the amount of  $H_2$  removed. Since the third stage acts as a  $H_2$  sensor, a separate  $H_2$  sensor in the  $N_2$  product stream is not required.
3. The Pd/Ag separator vents  $H_2$  to vacuum and requires an additional  $N_2$  sensor.

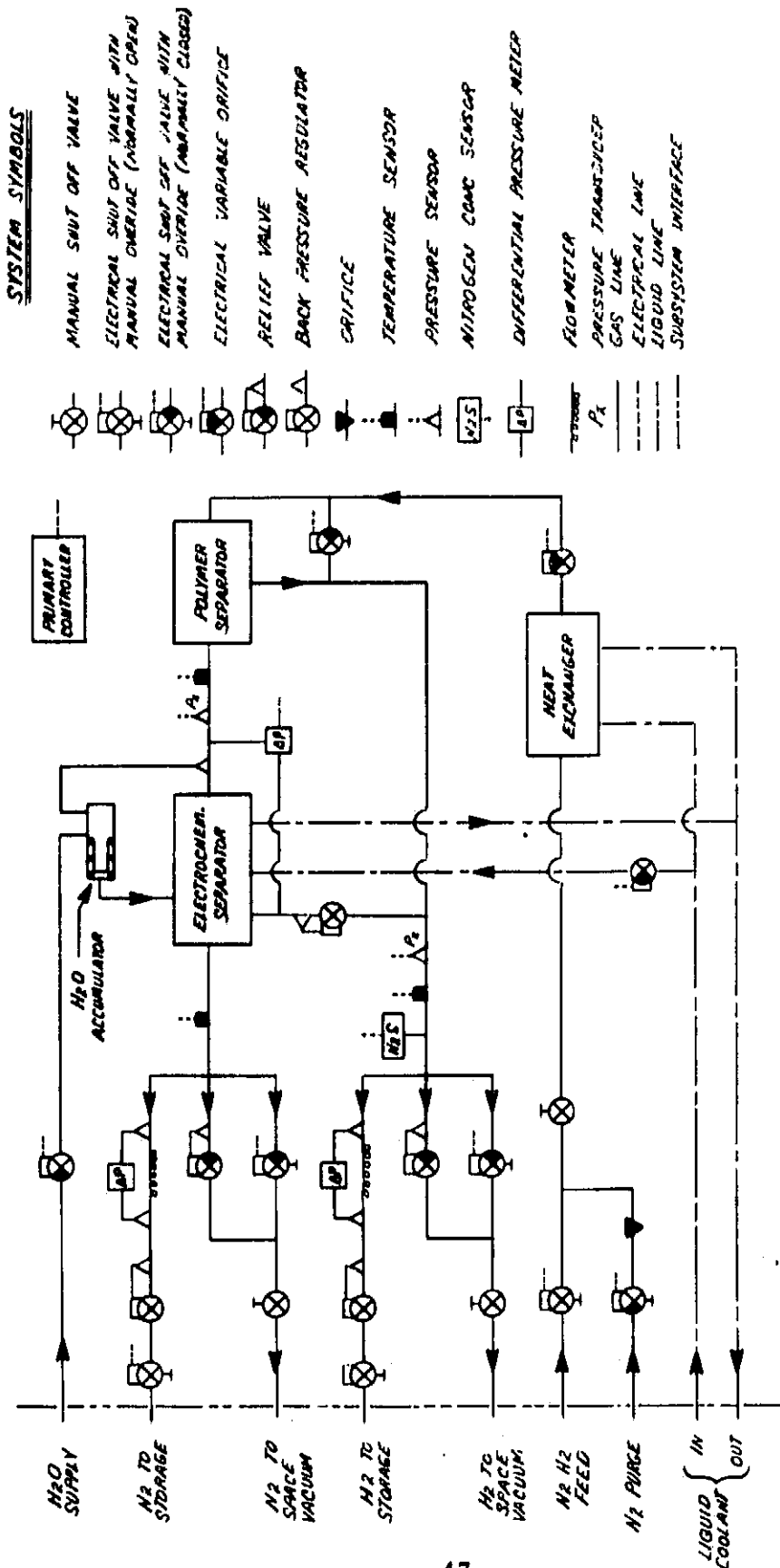


FIGURE 23 POLYMER-ELECTROCHEMICAL  $N_2/H_2$  SEPARATOR SUBSYSTEM SCHEMATIC



TABLE 7 COMPARISON OF THE Pd/Ag AND THE POLYMER-ELECTROCHEMICAL N<sub>2</sub>/H<sub>2</sub> SEPARATOR

	<u>Pd/Ag</u>	<u>Polymer-Electrochemical</u>
Separator Weight, kg (lb)	3.8 (8.3)	8.0 (17.6)
Power Required, W	0	17
Power Penalty <sup>(a)</sup> , kg (lb)	0	4.6 (10.1)
Heat Rejection, W	25	17
Heat Rejection Penalty, kg (lb)	5.0 (11.0) <sup>(b)</sup>	1.4 (3.1) <sup>(c)</sup>
Accessory Weight, kg (lb)	41.2 (90.8)	30.3 (66.8)
Accessory Power, W	269	269
Accessory Power Penalty <sup>(a)</sup> , kg (lb)	72 (159)	72 (159)
Accessory Heat Rejection, W	316	150
Accessory Heat Rejection Penalty <sup>(c)</sup> , kg (lb)	26.5 (58.4)	12.6 (27.8)
Packaging, kg (lb) at 50% of the basic system weight	74.3 (163.7)	64.5 (142.1)
Insulation, kg (lb) at 10% of the basic system weight	14.9 (32.8)	0
Spares Weight, kg (lb)	61.3 (135.0)	48.9 (107.7)
Spares Packaging, kg (lb) at 30%	18.4 (40.5)	14.7 (32.4)
Total Equivalent Weight, kg (lb)	317 (698)	257 (566)

(a) Continuous regulated DC at 0.268 kg/w (591 lb/kw).

(b) Heat rejected to cabin air at 0.198 kg/w (0.128 lb/Btu/hr).

(c) Heat rejected to liquid coolant at 0.084 kg/w (0.054 lb/Btu/hr).

4. Since the Pd/Ag separator system operates at elevated temperatures, components and gas lines require insulation.
5. The electrochemical stages require humidification and power to perform the separation.

#### Selection of the Optimum Approach

Selecting an optimum approach to  $N_2/H_2$  separation requires a closer evaluation of the polymer-electrochemical  $N_2/H_2$  Separator.

Based on preliminary estimates, the polymer-electrochemical  $N_2/H_2$  separator has the lowest total spared system equivalent weight for a spacecraft  $N_2$  Generator Subsystem. The integration of the polymer and electrochemical stages has not been demonstrated although no interfacing problems are envisioned. The logical step then is to design, fabricate, assemble and test a  $N_2/H_2$  separator system having a polymer gas permeator first stage and electrochemical second and third stages.

Further evaluation of the Pd/Ag system is not required since no advances in Pd/Ag technology are foreseeable.

#### CONCLUSIONS

The following conclusions are a direct result of this study.

1. A three-stage electrochemical  $N_2/H_2$  separator is not applicable to spacecraft  $N_2$  generation requirements because of the high power requirements of the first stage.
2. The power requirements of the second and third stages are applicable when combined with a passive first stage.
3. The moisture tolerance of the electrochemical cells is  $\pm 1K$  ( $\pm 2F$ ).
4. The use of internal humidifiers would increase the moisture tolerance and eliminate the need for an external  $N_2/H_2$  humidifier.
5. A full-sized second- and third-stage electrochemical  $N_2/H_2$  separator has been tested. No further scale-up is required.
6. A self-contained  $N_2/H_2$  separator for use in a nominal  $7.88 \times 10^{-5}$  kg/s (15 lb/day)  $N_2$  generator should be designed and tested using a polymer gas permeator as the first stage and an electrochemical separator as the second and third stages.

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